Effect of Thermal and Microwave Treatment on Low Temperature Mechanical Property of Glass Fiber Reinforced Polymer Composite: *An Experimental Exploration*

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CERTIFICATE

This is to certify that the report entitled, **'Effect of Thermal and Microwave Treatment on Low Temperature Mechanical Property of Glass Fiber Reinforced Polymer Composite:** ***An Experimental Exploration*** **'** submitted by **Prabhash Kumar Panda (107MM031)** and **Rakesh Nayak (107MM007)** in partial fulfillment of the requirements for the award of Bachelor of Technology Degree in Metallurgical & Materials Engineering at the National Institute Of Technology, Rourkela is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge, the matter embodied in the report has not been submitted to any other University/Institute for the award of any Degree or Diploma.

Date:

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CONTENTS

Abstract:

There has been a tremendous advancement in the science and technology of fiber reinforced polymer composites (FRP) in recent times. The low density, high strength, high stiffness to weight ratio, excellent durability, and design flexibility of fiber reinforced polymers are the primary reasons for their use in many structural components in the aircrafts, automotive, marine, transportation, sports, medical science and more recently the building and construction industries and particularly in areas that are weight and corrosion sensitive. A recent example is the Bridge-in-a-Backpack for 2014 Winter Olympics in Russia, an innovative inflatable composite-concrete arch bridge, which was developed to reduce construction time and costs, increase lifespan, reduce maintenance costs and reduce the carbon footprint of bridge construction[1]. But the phenomenon of the occurrence of their fracture and failure at low temperatures and under varying loading rates is a quite complex, and is not easily understandable phenomenon, because of the various types of the failure modes involved (e.g. delamination sites, debonding, fiber pullout regions, crack propagation front, striations and bubble bursting in the matrix). Thus a critical study has to be made to understand the overall phenomenon. Recently an active area of investigation related to this work is being explored by Temperature Modulated Differential Scanning Calorimetry (TMDSC) and Fourier Transform Infrared Spectroscopy (FTIR-Imaging), techniques to find out the possible causes for failure of the composite. In the present study, an attempt has been made for fractographic study of the composite material using SEM micrographs of the fractured surfaces of composites under Thermally Conditioned and post curing by Microwave treatment, followed by their exposure in ultralow temperatures, so that the origin of the crack could be analyzed and the factors affecting the locus of initiation of fracture could be determined. This would be followed by FTIR-Imaging and TMDSC to determine the alternation and deviation of Stoichiometry and the T_g values respectively, to have a better idea about the failure phenomenon.

Keywords: Fiber Reinforced Polymer Composites, Fiber Pullout, FTIR-Imaging, Fractography.

Chapter 1
INTRODUCTION

1.1 Composites

Composites are the 21st century material to meet the stringent demand of light weight, high strength and corrosion resistance shapes. Although composites were known to mankind since prehistoric times, the concept and technology have undergone a huge change with better understanding the basics like bonding mechanism between the matrix and fiber[2]. A composite is combination of two materials in which one of the materials, called the reinforcing phase, is in the form of fibers, sheets, or particles, and is embedded in the other materials called the matrix phase[3]. Typically, reinforcing materials are strong with low densities while the matrix is usually a ductile, or tough, material. The reinforcing material and the matrix material can be metal, ceramic, or polymer. In order to provide a useful increase in properties, there generally must be a substantial volume fraction (~10% or more) of the reinforcement.

1.1.1 Classification of Composites

Composites are commonly classified at two distinct levels. The first level of classification is usually made with respect to the matrix constituent. The major composite classes include organic-matrix composites (OMCs), metal-matrix composites (MMCs), and ceramic-matrix composites (CMCs). The term "organic-matrix composite" is generally assumed to include two classes of composites: polymer-matrix composites (PMCs) and carbon-matrix composites (commonly referred to as carbon-carbon composites). In each of these systems, the matrix is typically a continuous phase throughout the component.[4]

The second level of classification refers to the reinforcement form—particle reinforced, fiber reinforced, and structural composites.

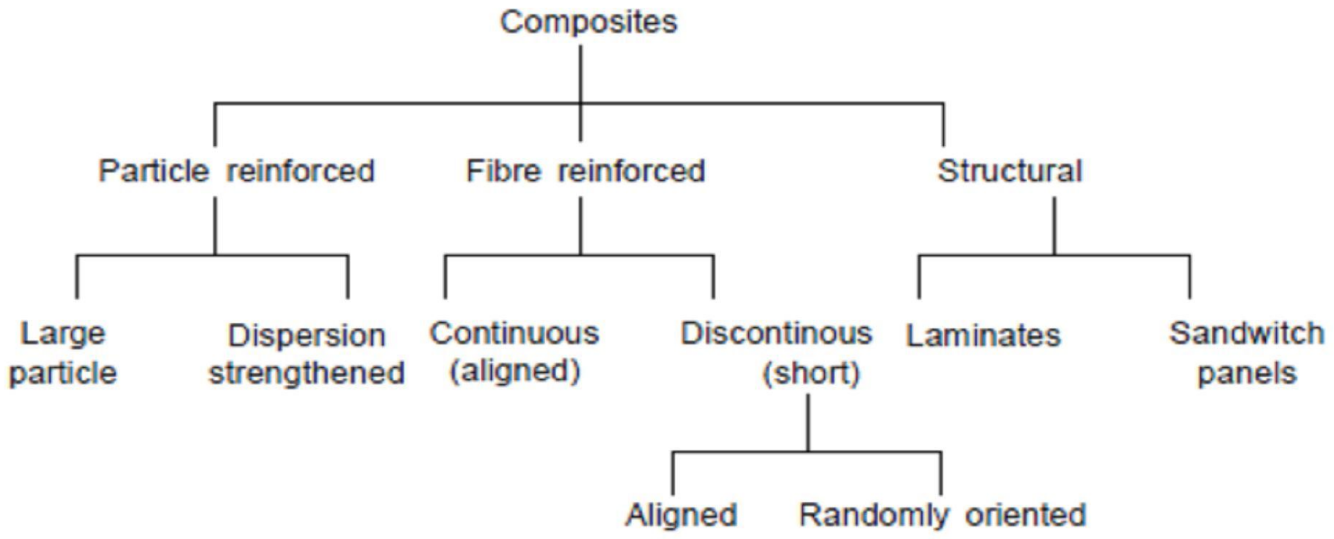


Fig 1.1 A simple classification schemes for the various composite types [5]

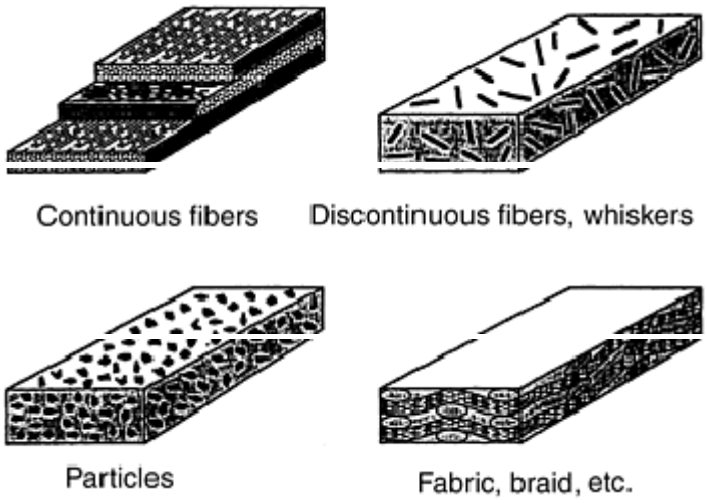


Fig 1.2 Types of fibers

1.1.2 Why Use FRP Composites

The following are some of the reasons why composites offer a potential advantages for certain applications:

- High strength to weight ratio (low density, high tensile strength)
- High creep resistance
- High tensile strength at elevated temperatures
- High toughness

A major advantage of GFRPs is that composite properties can be optimized for a specific application by varying the design factors, such as fiber volume content, fiber architecture, type of resin, and the chemical nature of the sizing applied to the surface of the fiber[6]. The higher strength of materials when they are converted to fibers has been the main driving force behind the development of composites. The mechanical properties of the components of the composite, i.e. the fiber, matrix and interphase, determine the mechanical behavior of composites on a macroscopic scale. The integrity of the composite as a whole depends upon the ease and effectiveness with which a load can be transferred within the composite[7].

1.1.3 Limitations of Composites

Susceptibility to de-lamination is one of the inherent weaknesses of laminated composite structures[8]. They are also susceptible to crack initiation and propagation along the laminar interfaces in various failure modes[9]. The fiber/matrix interface has always been considered as a crucial aspect of polymer composites. It is at the interface where stress concentration develops because of differences between the reinforcement and matrix phase thermal expansion coefficients. The interface may also serve as a locus of chemical reaction across which load is transferred and is of so such importance[10]. Another drawback of the thermoset resins is their tendency to absorb significant amounts of water when they are exposed to

hydrothermal environments. The temperature is likely to influence moisture pick-up kinetics in polymer composites in a complex manner[11].Due to cooling at ultra low temperatures, glass fibres exhibit longitudinal compressive stress. These stresses create thermal residual strain in the matrix. Compressive stresses developed in the fibre due to cooling are incorporated into the fibre failure strength distribution [12].

1.2. Glass Fibers

1.2.1 Types of Glass Fiber

The most common reinforcement for the polymer matrix composites is a glass fiber. Most of the fibers are based on silica (SiO_2), with addition of oxides of Ca, B, Na, Fe, and Al. The glass fibers are divided into three classes -- E-glass, S-glass and C-glass. The E-glass is designated for electrical use and the S-glass for high strength. The C-glass is for high corrosion resistance, and it is uncommon for civil engineering application. Of the three fibers, the E-glass is the most common reinforcement material used in civil structures. It is produced from lime-alumina borosilicate which can be easily obtained from abundance of raw materials like sand. The glass fiber strength and modulus can degrade with increasing temperature. Although the glass material creeps under a sustained load, it can be designed to perform satisfactorily. The fiber itself is regarded as an isotropic material and has a lower thermal expansion coefficient than that of steel.

- **E glass**

(Electrical)

Family of glassed with a calcium aluminum borosilicate composition and a maximum alkali composition of 2%. These are used when strength and high electrical resistivity are required.

- **S glass**

(Tensile strength)

Fibers have a magnesium alumino-silicate composition, which demonstrates high strength and used in application where very high tensile strength required.

- **C glass**

(Chemical)

It has a soda lime borosilicate composition that is used for its chemical stability in corrosive environment. It is often used on composites that contain or contact acidic materials.

- **R glass**

(Resistant)

R glass has a higher tensile strength and tensile modulus and greater resistance to fatigue, aging and temperature corrosion to that of E glass. [13]

1.2.2 Structure of Glass Fiber

Table 1.1 Composition of fibers

Composition of E-Glass

Constituent	Weight percentage
SiO2	54
Al203	14
CaO+MgO	12
B2O3	10
Na2O+K2O	Less than 2
Impurities	Traces

Comparison of properties of Glass Fiber

Typical Properties	E-Glass	S-Glass
Density (g/cm^3)	2.60	2.50
Young's Modulus (GPa)	72	87
Tensile Strength (GPa)	1.72	2.53
Tensile Elongation (%)	2.4	2.9

Glass fibers have high tensile strength, impact strengths and high chemical resistance. But these have relatively low modulus, self-abrasiveness, low fatigue resistance and poor adhesion to matrix composites.

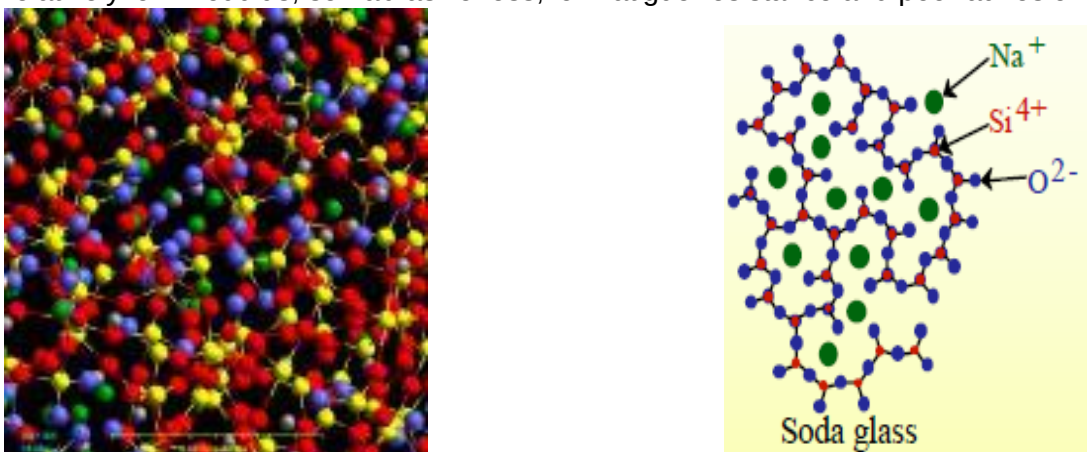


Fig 1.3 Polyhedra network structure of glass[14]

The three dimensional network of structure of glass results in isotropic properties of glass fibers, in contrast to those of Carbon and Kevlar aramid fibers which are anisotropic. The elastic modulus of glass fibers measured along the fiber axis is the same as that measured in the transverse direction, a characteristic unique to glass fibers[2].

1.3 Carbon fibers:

Carbon fiber, alternatively graphite fiber, is a material consisting of extremely thin fibers about 0.005–0.010 mm in diameter and composed mostly of carbon atoms. The carbon atoms are bonded together in microscopic crystals that are more or less aligned parallel to the long axis of the fiber. The crystal alignment makes the fiber very strong for its size. Several thousand carbon fibers are twisted together to form a yarn, which may be used by itself or woven into a fabric. Carbon fiber has many different weave patterns and can be combined with a plastic resin and wound or molded to form composite materials such as carbon fiber reinforced plastic (also referenced as carbon fiber) to provide a high strength-to-weight ratio material. The density of carbon fiber is also considerably lower than the density of steel, making it ideal for applications requiring low weight. The properties of carbon fiber such as high tensile strength, low weight, and low thermal expansion make it very popular in aerospace, civil engineering, military, and motorsports, along with other competition sports. However, it is relatively expensive when compared to similar materials such as fiberglass or plastic. Carbon fiber is very strong when stretched or bent, but weak when compressed or exposed to high shock. Carbon fibers can be classified depending on the precursor used, the most commonly used precursors are rayon based fibers, polyacrylonitrile (PAN) and pitch.

Carbon-fiber-reinforced polymer (CFRP), is a very strong, light, and expensive composite material or fiber-reinforced polymer. The polymer is most often epoxy, but other polymers, such as polyester, vinyl ester or nylon, are sometimes used[15].

Fig 1.4 Carbon Fibres

1.4 Kevlar fibers:

Aramid fibers are a class of heat-resistant and strong synthetic fibers. They are used in aerospace and military applications, for ballistic rated body armor fabric and ballistic composites, in bicycle tires, and as an asbestos substitute. The name is a portmanteau of "aromatic polyamide". They are fibers in which the chain molecules are highly oriented along the fiber axis, so the strength of the chemical bond can be exploited. Currently, Kevlar has many applications, ranging from bicycle tires and racing sails to body armor because of its high tensile strength-to-weight ratio; by this measure it is 5 times stronger than steel on an equal weight basis. When used as a woven material, it is suitable for mooring lines and other underwater applications.

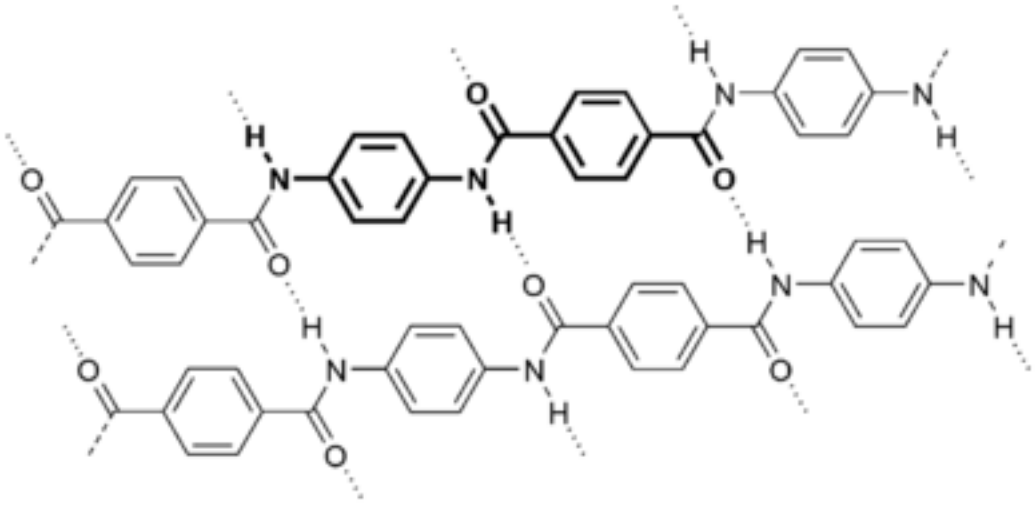


Fig 1.5 Schematic representation of repeat unit and chain structures for Kevlar fiber[15]

1.5 Epoxy Resins

Epoxy resins are relatively low molecular weight pre-polymers capable of being processed under a variety of conditions. Two important advantages of these resins are over unsaturated polyester resins are: first, they can be partially cured and stored in that state, and second they exhibit low shrinkage during cure. Approximately 45% of the total amount of epoxy resins produced is used in protective coatings while the remaining is used in structural applications such as laminates and composites, tooling, moulding, casting, construction, adhesives, etc[13].

Epoxy resins are characterized by the presence of a three-membered ring containing two carbons and an oxygen (epoxy group or epoxide or oxirane ring). Epoxy is the first liquid reaction product of bisphenol-A with excess of epichlorohidrin and this resin is known as diglycidylether of bisphenol A (DGEBA). DGEBA is used extensively in industry due to its high fluidity, processing ease, and good physical properties of the cured of resin.

Epoxy is a copolymer; that is, it is formed from two different chemicals. These are referred to as the "resin" and the "hardener". The resin consists of monomers or short chain polymers with an epoxide group at either end. Most common epoxy resins are produced from a reaction between epichlorohydrin and bisphenol-A, though the latter may be replaced by similar chemicals. The hardener consists of polyamine monomers, for example Triethylenetetramine1. When these compounds are mixed together, the amine groups react with the epoxide groups to form a covalent bond. Each NH group can react with an epoxide group, so that the resulting polymer is heavily cross linked, and is thus rigid and strong[16].

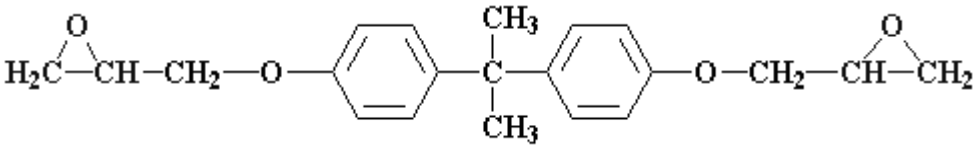


Fig 1.6 Structure of DGEBA

Ethylene diamines are most widely used aliphatic amines for cured epoxy resins. These are highly reactive, low molecular weight curing agents that result in tightly cross-linked network. One primary amino group reacts with two epoxy groups. The primary and secondary amines are reactive curing agents. The primary amino group is more reactive towards epoxy than secondary amino groups are consumed (95%), whereas only 28% of secondary amino groups are consumed

The process of polymerization is called "curing", and can be controlled through temperature, choice of resin and hardener compounds, and the ratio of said compounds; the process can take minutes to hours. Some formulations benefit from heating during the cure period, whereas others simply require time, and ambient temperatures.

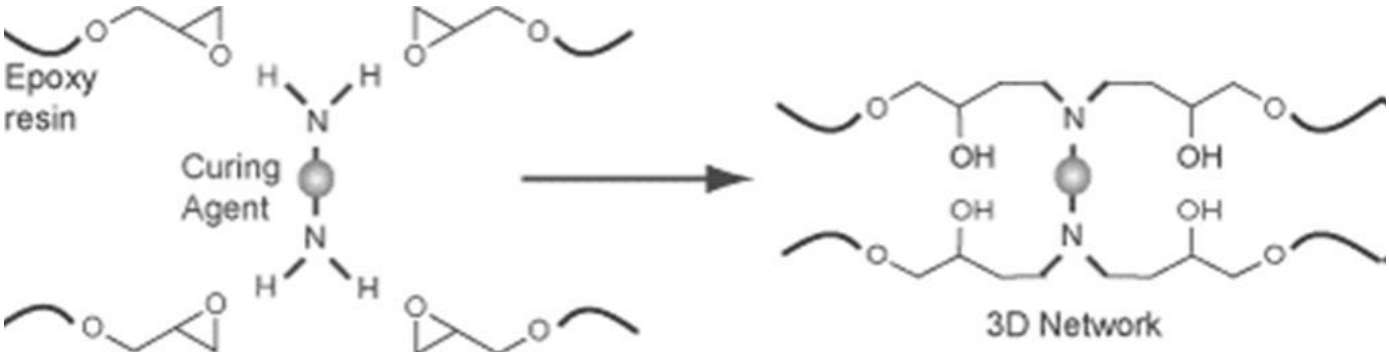


Fig 1.7 Polymerization of DGEBA during curing[14]

Chapter 2
LITERATURE SURVEY

2.1 Interfaces and Interphases in Composites:

The word “interphases” refers to a region where the fiber and matrix phases are chemically and /or mechanically combined or otherwise indistinct[17]. An “interface” is a boundary demarcating distinct phases such as fiber, matrix, coating layer, or interphase[13].

Usually, it is accepted that the interphase region has a thickness of 100 to 500 nm[18].

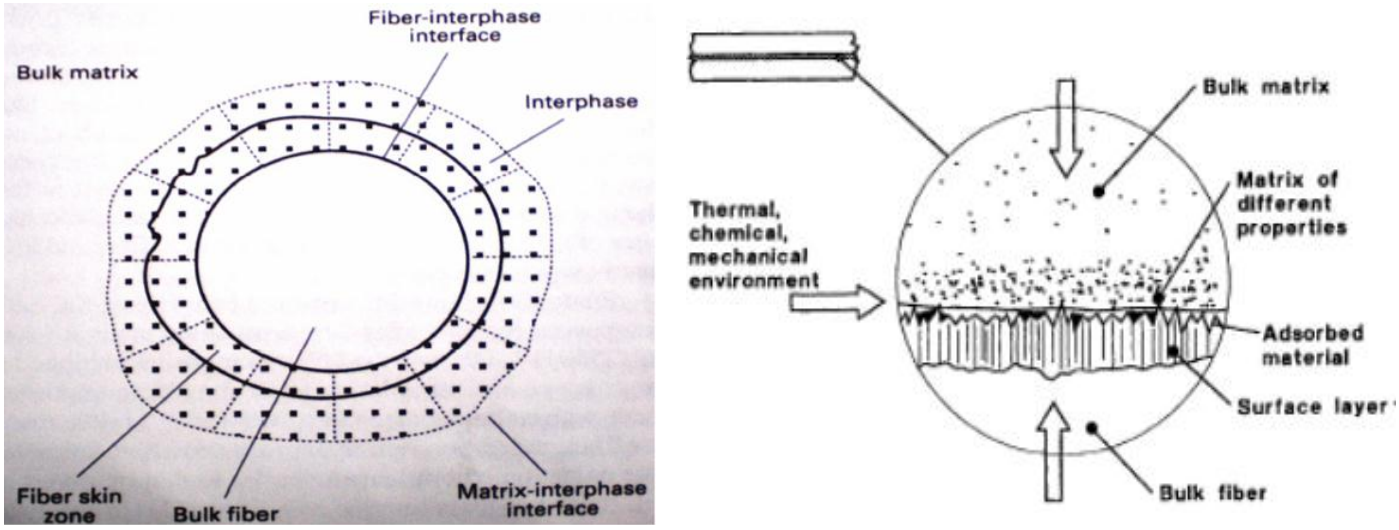


Fig.2.1 Fiber-matrix interface and interphase[13]

Advanced composites cannot be manufactured with good performance without optimum adhesion and appropriate interphases[19]. Interfacial interactions and interphases play a key role in all multicomponent materials irrespectively of the number and type of their components or their actual structure. They are equally important in particulate filled polymer, polymer blends, fiber reinforced advanced composites, nanocomposites or biomaterials. Although the role and importance of interfaces and interphases are the same for all multicomponent materials, surface modification must be always selected according to the objectives targeted, as well as to the characteristics of the particular system[20].Thus the integrity of the composite as a whole depends upon the ease and effectiveness with which a load can be transferred within the composite. Thus the interface, i.e. the boundary across which load is transferred serves as the heart of composite, which implies, the mechanical behavior of a composite material is decisively controlled by the fiber-matrix interface.

2.2 The Failure Analysis

Fractographic techniques can be used to study micro-mechanisms of fracture, investigate of failure in lab oratory structures, and post-mortem investigation of in-service components. The basic approach is to characterize the fracture morphologies of specimens failed under known (pure) failure modes, and then compare these morphologies to 'unknown' failures.
In composites the main causes of failure can be:

1. De-lamination.
2. Interfacial De-bonding (separation of fibers &matrix).
3. Microcracking of the matrix.
4. Fiber pull-out
5. Breaking of fibers
6. Stress redistribution
7. Longitudinal matrix splitting

Fracture modes in composites can be divided into three basic fracture types
a) Interlaminar
b) Intralaminar
c) Translaminar

When considered on microscale, interlaminar and intralaminar fracture types can be similarly described. In both cases, fracture occurs on a plane parallel to that o f the fiber reinforcement. In a similar manner to that described for metals ,fracture of either type can occur under mode I tension, mode II in-plane shear, mode III anti- plane shear, or any combination of these load conditions. Translaminar fractures are those oriented transverse to the laminated plane in which conditions of fiber fractures are generated.
It is possible to split the failure models developed on statistical bases in relation to fibre-reinforced composites into two categories: the weakest link model and fracture models. The weakest link model also known as the series model is based on the assumption that the whole structure fails if the weakest link fails. But, the series model does not apply to composite materials, since the unbroken fibres continue to carry loads after the weakest fibres break.
Turning to the fracture models, two major models are presented in the literature: the cumulative fracture model and the fracture propagation model. In the first model, the matrix is assumed not to contribute directly to the tensile strength of the composites, although it provides a means to transfer the load in shear

to the fibres. The specimen is divided into layers (bundles) of a length defined as an ineffective length ~0. When the specimen is loaded, the fibres are assumed to be stressed uniformly and as the load increases the fibres in each bundle start to break randomly and stresses are redistributed uniformly among the unbroken fibres in each bundle. When a sufficient number of fibres in a bundle fail, the specimen fails.[12]

2.2.1 Delamination

Delamination is a mode of failure for composite materials[21]. Modes of failure are also known as 'failure mechanisms'. In laminated materials, repeated cyclic stresses, impact, or 3-point loading can cause layers to separate, with significant loss of mechanical toughness. Delamination also occurs in reinforced concrete structures subject to reinforcement corrosion, in which case the oxidized metal of the reinforcement is greater in volume than the original metal. The oxidized metal therefore requires greater space than the original reinforcing bars, which causes a wedge-like stress on the concrete. This force eventually overcomes the relatively weak tensile strength of concrete, resulting in a separation (or delamination) of the matrix above and below the reinforcing fibers[15].

The cause of fiber pull-out (another form of failure mechanism) and delamination is weak bonding[22]. Thus, delamination is an insidious kind of failure as it develops inside of the material, without being obvious on the surface, much like metal fatigue. Delamination is a critical failure mode in composite structures as can degrade the laminate to such a degree that it becomes useless in service. The interfacial separation caused by the delamination may lead to premature buckling of the laminate, excessive vibration, intrusion of moisture, stiffness degradation and loss of fatigue life . The delamination though in some cases may provide stress relief and actually enhance the performance of the component[13].

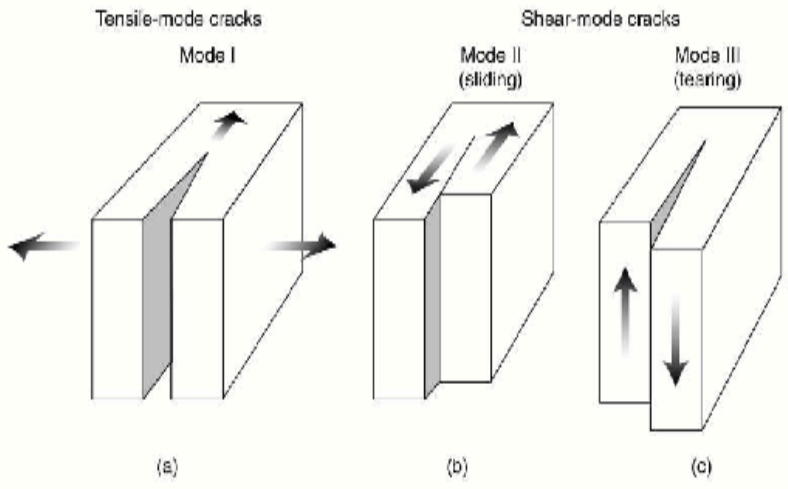


Fig 2.2 Crack Opening Modes

Delamination may be introduced during processing or in service conditions. It may result from low velocity impact, from eccentricities in the structural load.

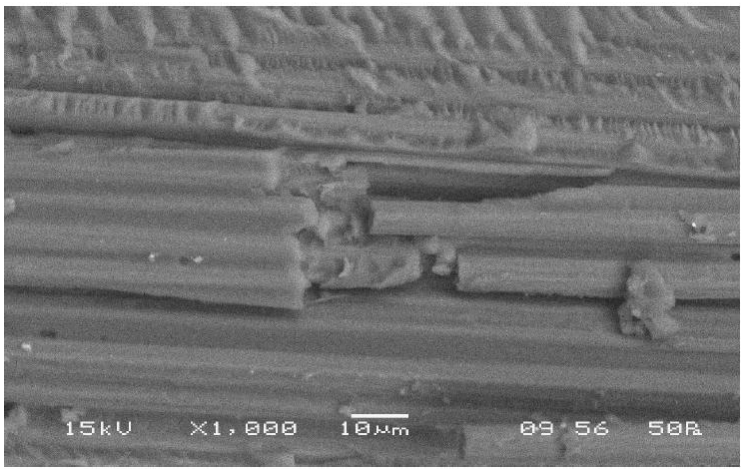


Fig 2.3 Delamination[23]

In addition to mechanical loads, the moisture and temperature may also induce interlaminar stresses in a laminate. These may be the results from the residual thermal stresses caused from cooling from processing temperatures and residual stresses created by the absorption of moisture. The delamination may lead to redistribution of stresses which would eventually promote gross failure.

2.2.2 Fiber Pull Out and Debonding:

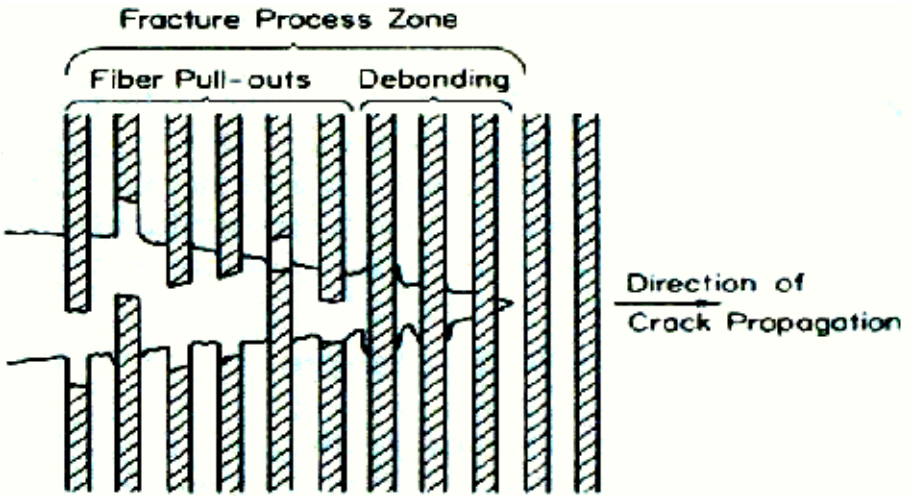
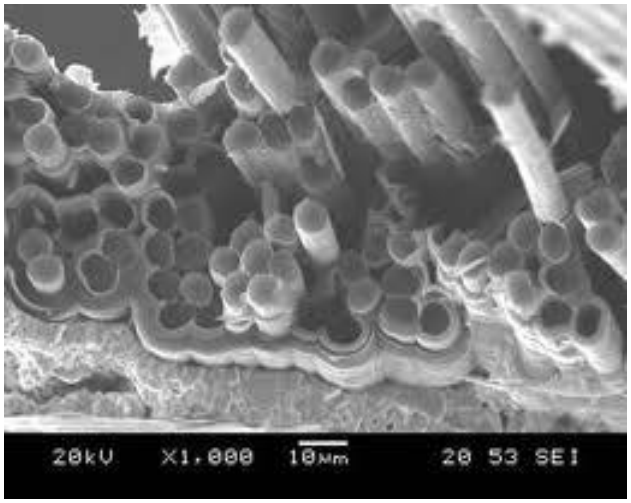


Fig 2.4 Crack tip showing local failure events.

At some distance ahead of the crack the fibers are intact. In the high stress region near the tip, they are broken, not necessarily along the crack plane. Immediately behind the crack tip fibers pull out of the matrix. In some composites the stress near the crack tip could cause the fibers to debond from the matrix before they break. When brittle fibers are well bonded to a ductile matrix, the fibers tend to snap ahead of the crack tip, leaving bridges of matrix material that neck down and fracture in a

completely ductile manner. In addition to these local failure mechanisms, on reaching the interface of the two laminate in a laminated.



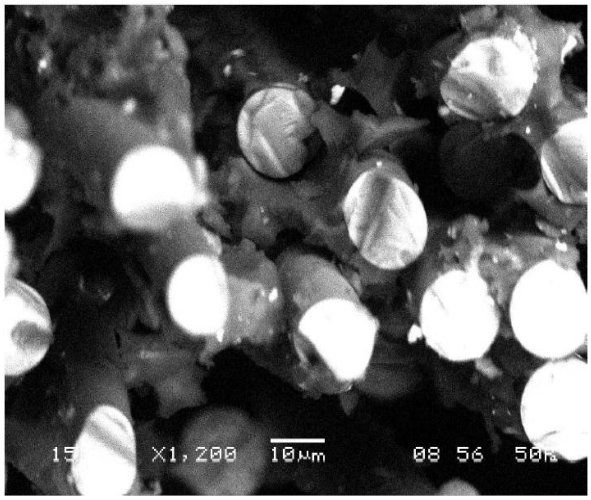


Fig 2.5 Fiber Pull Out[24]

In fibre reinforced materials with both brittle fibers and brittle matrices, toughness is derived from two sources. Firstly, if the crack can be made to run up and down every fibre in its path the there will be a large amount of new surface created for a very small increase in crack area perpendicular to the maximum principal stress - Interfacial Energy - and in order to get the fibers to break they have to be loaded to their fracture strength and this often requires additional local elastic work, and secondly if the fibers do not break and therefore bridge the gap then work must be done to pull the fibers out of the matrix - Fibre Pullout. Using simple geometric models we can estimate the contribution of each of these processes to the overall toughness of the composite[25].

2.2.3 Matrix Microcracking

The first form of damage in laminates is often matrix micro cracking. They are intralaminar or ply cracks that traverse the thickness of the ply and run parallel to the fibers of the ply[26]. The most common observable micro cracking is cracking in the 900 plies during axial loading in the direction. These micro cracks are transverse to the loading direction and are often termed as the transverse cracks. Tensile loading, fatigue loading, environment, and thermal cycling can all lead to microcrack formation[27]. Microcracks can form in any ply that has a significant component of the applied load transverse to the fibers in that ply. Microcracks lead to degradation in properties of the laminate including changes in effective moduli, Poisson ratios, and thermal expansion coefficients[28]. Although these changes are sometimes small, microcracks can nucleate other forms of damage.

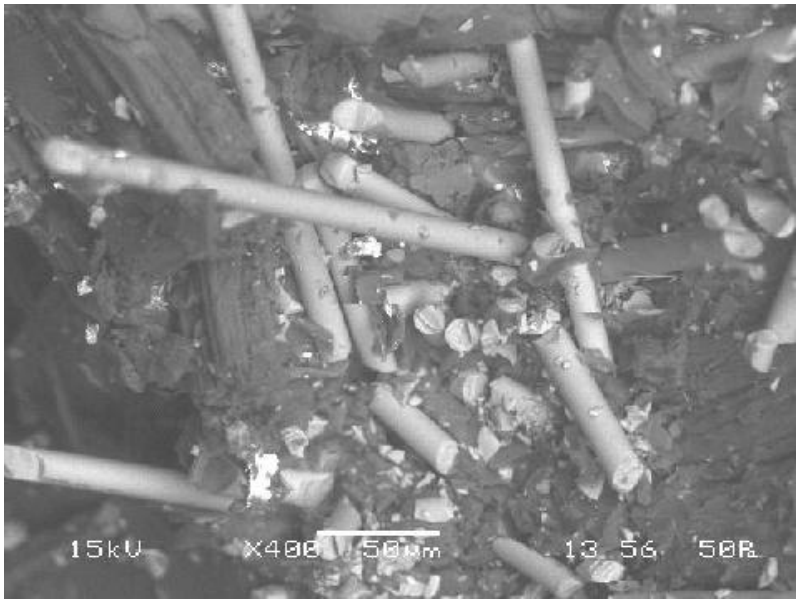


Fig 2.6 Matrix Microcracking[29]

2.2.4 Fiber Fracture:

If the amount of stress being applied is unable to be sustained by the fibers when distributed on them by the matrix due to the inability of the formation of strong interfacial bonds, breaking of fibers may occur. This can take place if the glass fiber is aptly held by the matrix and will be followed by the rupture of the fibers which may be due to the localized stress and strain fields in the fibrous composite.

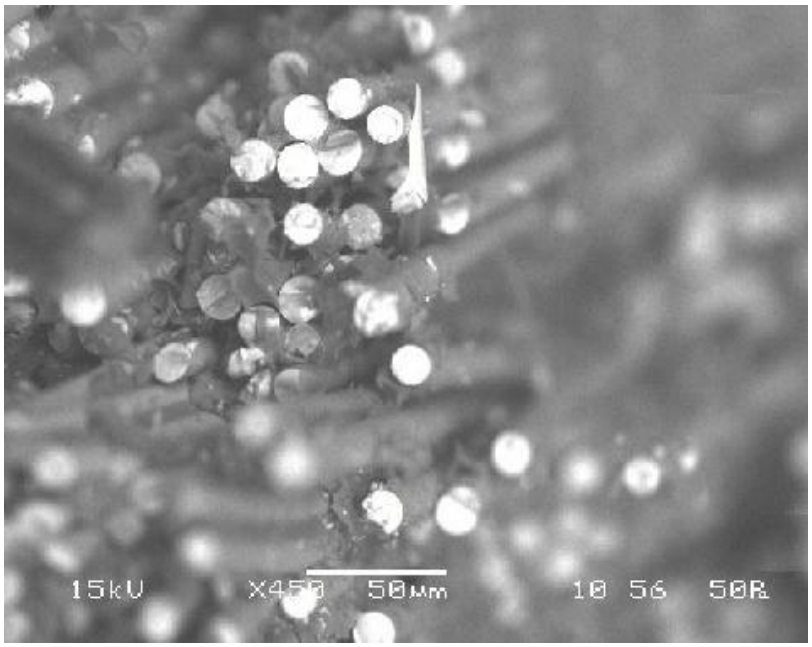


Fig 2.7 Breaking of Fibres[29]

2.3 Effect of Ultra Low Temperatures

The resulting properties on exposure of fibre reinforced composites to liquid nitrogen are strongly dependent on the factors such as the matrix and fibre material and their volume fractions, the fibre orientation, the applied stress levels and strain rates, as well as the loading conditions and the nature of fibre polymer interface. Interface is said to be the heart of the composite[30]. The local response of fibre matrix interface within the composite plays an important role in determining the gross mechanical performance. It provides a means of stress transfer from fibre to fibre through the matrix. In cold conditions, high residual stresses can build up within the fibrous composite materials due to different coefficients of thermal expansion of the fibre and the matrix and at low temperatures the polymer matrix experiences embrittlement which can also affect the properties of the composite[31].

Potholing or localized surface degradation, delamination, and microcracking are some of the more dramatic phenomena that can occur as a result of cryogenic cycling. Increased thermal stresses are the underlying cause of micro cracking in composites at cryogenic temperatures. As the laminate temperature falls below its stress free temperature, residual stresses develop in the material. These stresses are the result of a difference in the linear coefficient of thermal expansion (CTE) between the fibers and the matrix [32]. The generated residual stresses influence the overall thermo-mechanical properties of the composite. In some cases, the resulting stresses are sufficient to initiate plastic deformation within the matrix immediately around the fiber. Therefore, it is important to determine the current state of the residual stresses and their effects on the behavior of the composite when subsequently subjected to various uniaxial and multiaxial mechanical loading. The stresses can also be large enough to initiate material damage such as matrix micro cracking. These micro cracks can reduce the strength of the material, as well as act as sites for environmental degradation and nucleation of macro cracks[33].

2.4 Effect of Microwave Radiation:

Microwave heating involves direct energy absorption by the material; consequently, it is possible to heat a polymer matrix more uniformly and rapidly than with conventional heating using an electrically heated oven, which relies on the thermal conductivity of such materials. Also, with microwave heating, the heating rate can be instantaneously controlled either by changing the incident microwave power, or by on/off switching of the microwave field, or both. As a result, the temperature of a polymerizing sample can be more effectively controlled during critical periods, such as generation of exothermic heat of

polymerization[49]. Microwaves heat from the inside out as each molecule is generating heat from "inside" and radiating it "outward".

Fig 2.8 A combined microwave and fan-assisted oven, with the door opened.

A microwave oven works by passing non-ionizing microwave radiation, usually at a frequency of 2.45 gigahertz (GHz)—a wavelength of 122 millimetres (4.80 in)—through the material. Microwave radiation is between common radio and infrared frequencies. The material absorbs energy from the microwaves in a process called dielectric heating. Many molecules that may act as electric dipoles, i.e., they have a partial positive charge at one end and a partial negative charge at the other, and therefore rotate as they try to align themselves with the alternating electric field of the microwaves. This molecular movement represents heat which is then dispersed as the rotating molecules hit other molecules and put them into motion.

Microwave heating can cause localized thermal runaways in material with low thermal conductivity, where dielectric constant increases with temperature. Under certain conditions, glass can exhibit thermal runaway in a microwave to the point of melting[15].

2.5 Effect of Thermal Aging:

Thermal degradation of epoxy resin involves chemical reaction and physical changes. Chemical reaction is represented by oxidation, further cross-linking and further reaction of un-reacted monomers, while physical change is the viscoelastic behavior. The visco-elastic yield behavior of polymer is generally temperature and loading rate dependent. The thermal conditioning results in post-curing strengthening effect. Thermal aging behavior of epoxy resins is of special interest because of their expanding use for structural applications where increased temperatures are common environmental conditions. Fiber reinforced composites are sensitive to temperature variations as a result of the build-up of thermal stresses between the fibres and binder due to their distinct thermal expansion coefficients. The induced thermal stresses may be relieved by crack formation in the matrix and, in extreme cases, by fiber failure[34].

2.6 Effect of Loading Rate:

Composite structures undergo different loading conditions during their service life, e.g. sports equipment at high loading rate to pressure vessels at low loading rates [35]. Composites are being used increasingly in applications where they are deformed rapidly. One of these applications is when composite jet engine compressor blades are exposed to hazards of foreign object damage, such as bird impact on rotating blades, this impact occurring at velocities of up to 300 m s^{-1} and being able to cause extensive damage to the composite blade[36]. The effects of varying loading rates on mechanical properties of FRP composites are investigated and observed a variety of contradictory observations and conclusions [37]. E-Glass fibers have been found to be rate sensitive, information available in literature was not extensive to draw any concrete conclusion. Woven and unidirectional GFRP are rate dependent, both the modulus and strength increase as the test rate are increased, strain to failure decrease with increasing strain rate [38, 39, 40].

2.6 Temperature Modulated Differential Scanning Calorimetry:

Differential scanning calorimetry (DSC) is a technique for measuring the energy necessary to establish a nearly zero temperature difference between a substance and an inert reference material, as the two specimens are subjected to identical temperature regimes in an environment heated or cooled at a controlled rate[42].

A new advanced technique of temperature-modulated DSC (TMDSC) combines the separation of sensible and latent heat flow and the measurement of the frequency-dependent heat capacity over a wide frequency range. This technique is based on stochastic temperature modulation and yields the quasi-static heat capacity and the frequency-dependent complex heat capacity without the need for additional calibration procedures. Thus, it can be determined over a wide frequency range. A second result of the analysis is the non-reversing heat flow. This is the non-correlated heat flow component. The reversing heat flow is calculated from the quasi-static heat capacity. All these quantities and their frequency dependency can be determined in one single measurement[15].

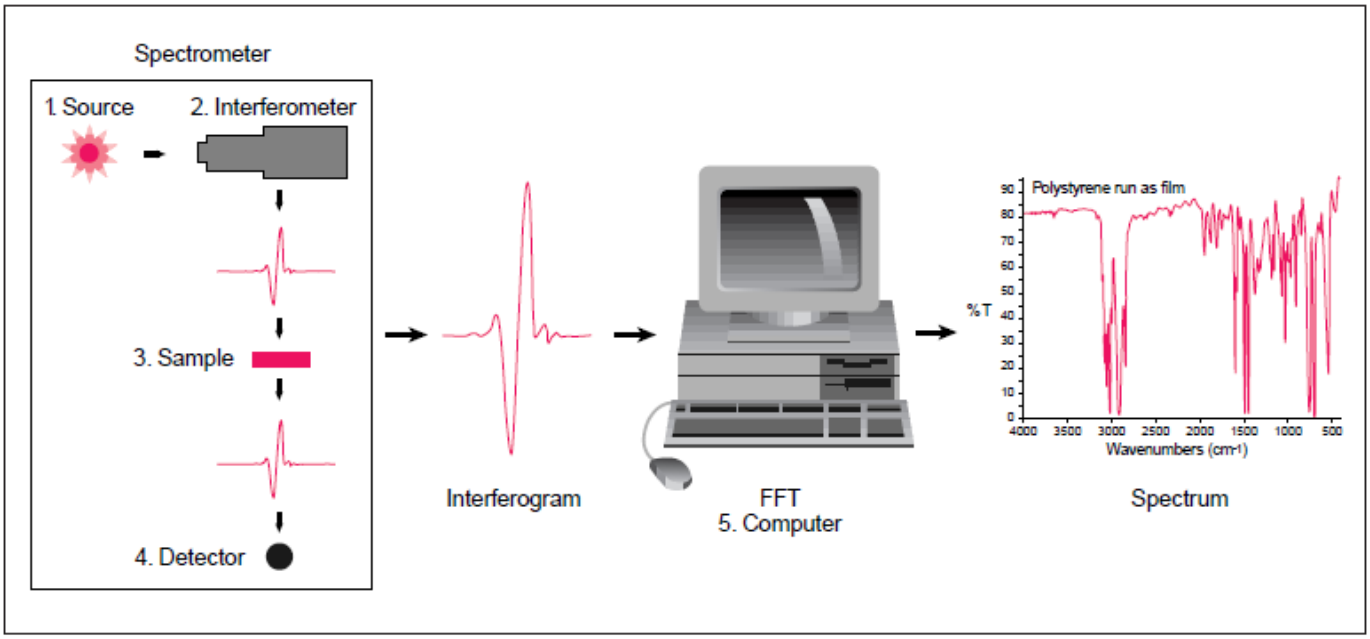
2.7 FTIR Imaging

Fourier transform infrared spectroscopy (FTIR) is a technique which is used to obtain an infrared spectrum of absorption, emission, photoconductivity or Raman scattering of a solid, liquid or gas. An FTIR spectrometer simultaneously collects spectral data in a wide spectral range. This confers a significant

advantage over a dispersive spectrometer which measures intensity over a narrow range of wavelengths at a time. FTIR technique has made dispersive infrared spectrometers all but obsolete (except sometimes in the near infrared) and opened up new applications of infrared spectroscopy.

The term Fourier transform infrared spectroscopy originates from the fact that a Fourier transform (a mathematical algorithm) is required to convert the raw data into the actual spectrum[43].

FTIR is most useful for identifying chemicals that are either organic or inorganic. It can be utilized to quantitate some components of an unknown mixture. It can be applied to the analysis of solids, liquids, and gasses. The term Fourier Transform Infrared Spectroscopy (FTIR) refers to a fairly recent development in the manner in which the data is collected and converted from an interference pattern to a spectrum. Today's FTIR instruments are computerized which makes them faster and more sensitive than the older dispersive instruments[15].

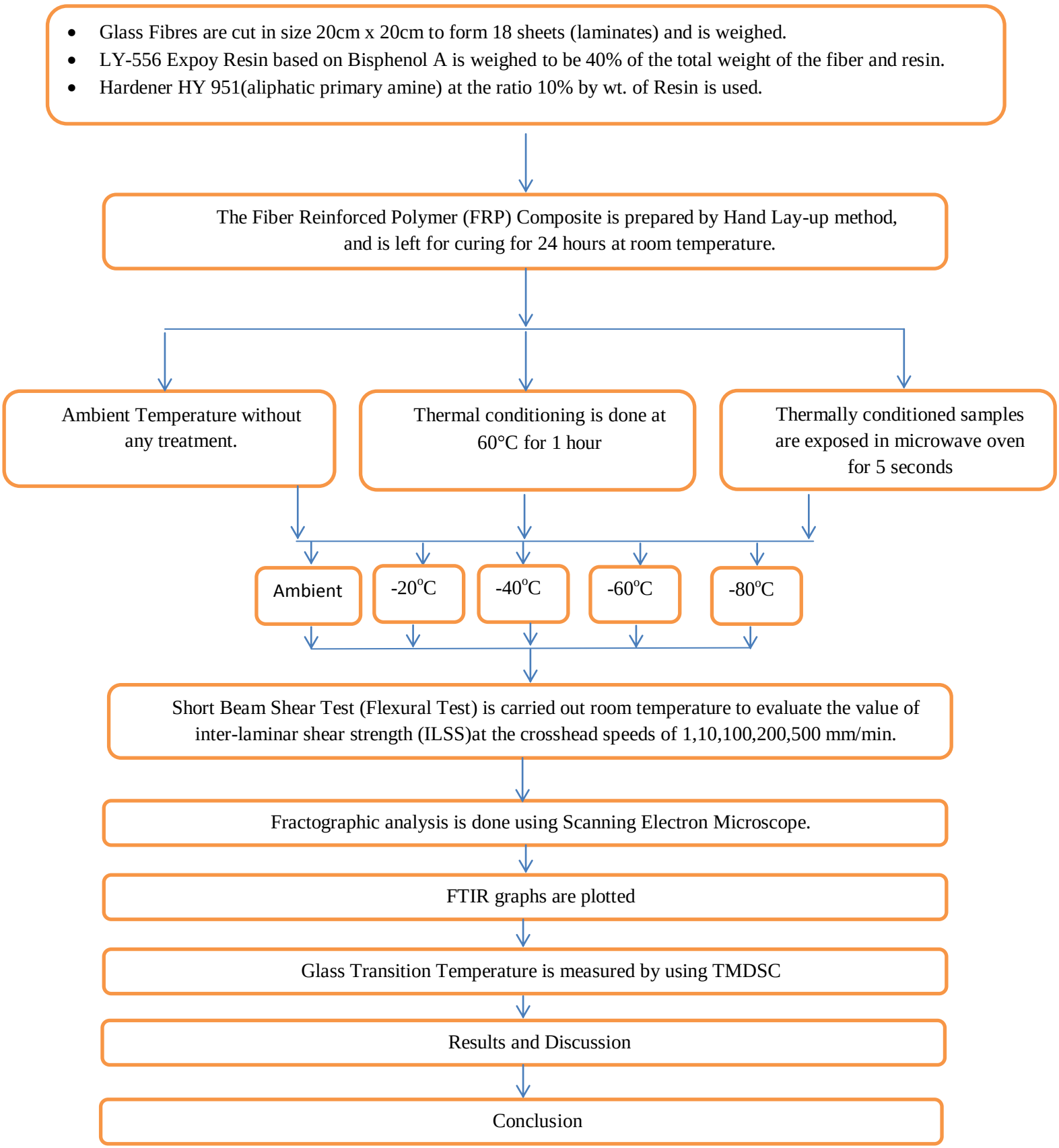


The goal of FTIR is to measure how well a sample absorbs light at each wavelength. The most straightforward way to do this, the "dispersive spectroscopy" technique, is to shine a monochromatic light beam at a sample, measure how much of the light is absorbed, and repeat for each different wavelength.

Fourier transform spectroscopy is a less intuitive way to obtain the same information. Rather than shining a *monochromatic* beam of light at the sample, this technique shines a beam containing many different frequencies of light at once, and measures how much of that beam is absorbed by the sample. Next, the beam is modified to contain a different combination of frequencies, giving a second data point. This process is repeated many times. Afterwards, a computer takes all these data and works backwards to infer what the absorption is at each wavelength[44].

Chapter 3
Experimental

3.1 Experimental work :.



Chapter 4
Results and Discussions

Thermal conditioning of the composite increases interfacial bonding, thereby leading to further polymerization due to higher degree of cross linking. The penetrating and semi penetrating network leads to further cross linking due to enhanced adhesion at the interface. But due to thermal conditioning, the matrix may also get brittle and may be prone to crack initiation.

Treatment in microwave radiation leads to homogenous heating as heat traverses from core to surface, so there may be more uniformly cross linking. This in turn exposes more matrix for further polymerization, and thus more penetrating and semi penetrating offset.

When Glass Fiber Reinforced Polymers are exposed to ultra low temperatures, the prominent damaging mechanisms may be matrix cracking, matrix hardening, de-bonding and damage due to difference in thermal coefficient of fiber and matrix.

The variation in Crosshead Speed can give us an idea about the damaging propensity of the composite towards the alterations in different loading rates. Generally, increase in loading rate results in improved interfacial chemistry. So, by this variation, we can estimate the change in locus of failure.

4.1 Short Beam Shear Testing:

4.1.1 ILSS Values

After short beam shear test at the conditioning temperature of the samples,

The ILSS values are calculated as:

ILSS = 0.75P/bd

Where, P is maximum load, b the width of specimen and d the thickness of specimen

Table 4.1:The ILSS values for the samples at ambient atmosphere

Loading rates	Ambient	Heat treated at 60°C	Microwave post heat treatment
1	23.81	20.62	22.61
10	23.1	25.14	22.73
100	29.7	17.31	18.94
200	20.7	22.89	24.42
500	23.59	23.86	21.74

Table 4.2: The ILSS values For Samples at -20°C

Loading rates	Ambient	Heat treated at 60°C	Microwave post heat treatment
1	23.46	23.9	21.31
10	24.38	24.65	27.46
100	21.84	22.8	26.05
200	21.78	23.31	21.27
500	24.51	22.07	20.63

Table 4.3: The ILSS values For Samples at -40°C

Loading rates	Ambient	Heat treated at 60°C	Microwave post heat treatment
1	23.49	22.23	26.65
10	20.45	22.61	23.23
100	21.91	24.31	22.02
200	21.43	24.65	20.89
500	21.77	20.99	20.95

Table 4.4: The ILSS values For Samples at -60°C

Loading rates	Ambient	Heat treated at 60°C	Microwave post heat treatment
1	21.3	18.95	22.94
10	21.51	23.83	24.03
100	22.14	22.7	24
200	19.08	20.02	21.15
500	20.9	23.37	21.52

Table 4.5: The ILSS values For Samples at -80^{o}C

Loading rates	Ambient	Heat treated at 60^{o}C	Microwave post heat treatment
1	19.46	21.01	23.64
10	17.95	23.56	24.5
100	14.32	23.31	22.43
200	17.76	19.88	20.37
500	20.08	21.47	21.89

Alternatively,

Table 4.6:The ILSS values under Ambient Conditions at varying temperatures

Loading rates	Ambient	-20 C	-40 C	-60 C	-80 C
1	23.81	23.46	23.49	21.3	19.46
10	23.1	24.38	20.45	21.51	17.95
100	29.7	21.84	21.91	22.14	14.32
200	20.7	21.78	21.43	19.08	17.76
500	23.59	24.51	21.77	20.9	20.08

Table 4.7: The ILSS values under Heat treated conditioned at 60^{o}C at varying temperatures

Loading rates	Ambient	-20 C	-40 C	-60 C	-80 C
1	20.62	23.9	22.23	18.95	21.01
10	25.14	24.65	22.61	23.83	23.56
100	17.31	22.8	24.31	22.7	23.31
200	22.89	23.31	24.65	20.02	19.88
500	23.86	22.07	20.99	23.37	21.47

Table 4.8: The ILSS values under Microwave post heat treatment conditioned at varying temperatures

Loading rates	Ambient	-20 C	-40 C	-60 C	-80 C
1	22.61	21.31	26.65	22.94	23.64
10	22.73	27.46	23.23	24.03	24.5
100	18.94	26.05	22.02	24	22.43
200	24.42	21.27	20.89	21.149	20.37
500	21.74	20.63	20.95	21.52	21.89

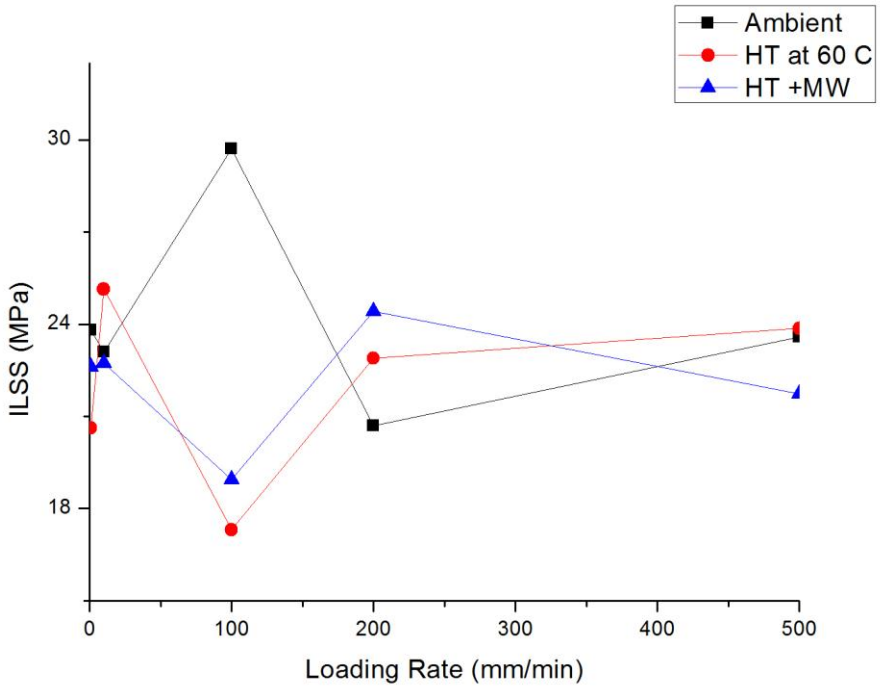


Fig 4.1: ILSS Vs Crosshead speed for Glass/epoxy composites at ambient temperature

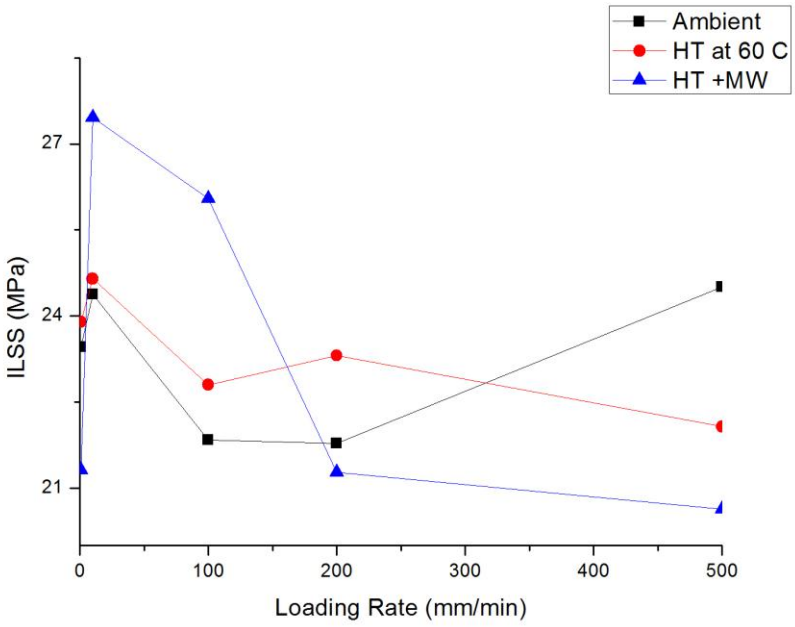


Fig 4.2: ILSS Vs Crosshead speed for Glass/epoxy composites at -20^{o}C

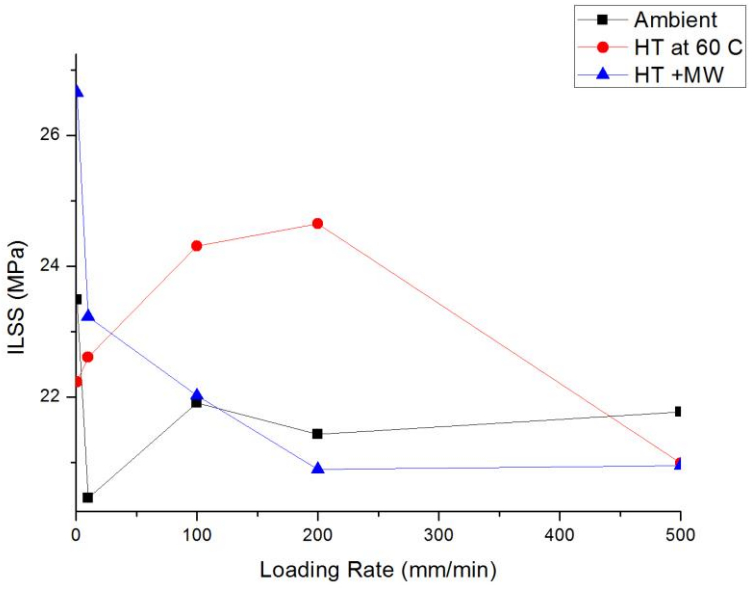


Fig 4.3: ILSS Vs Crosshead speed for Glass/epoxy composites at -40^{o}C

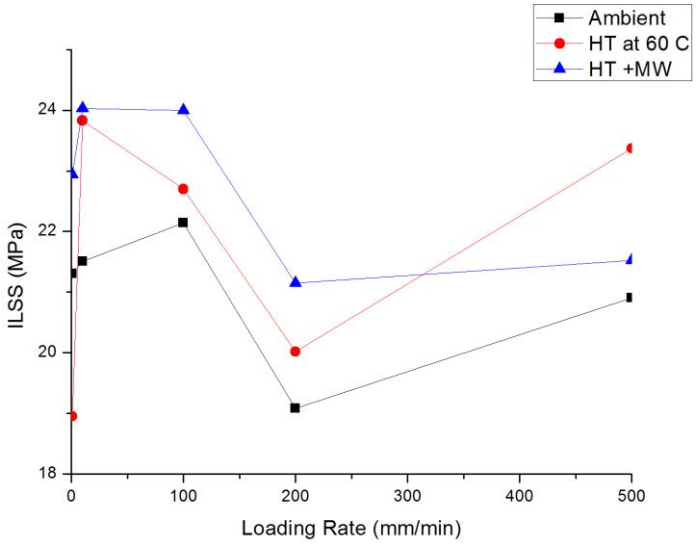


Fig 4.4: ILSS Vs Crosshead speed for Glass/epoxy composites at -60^{o}C

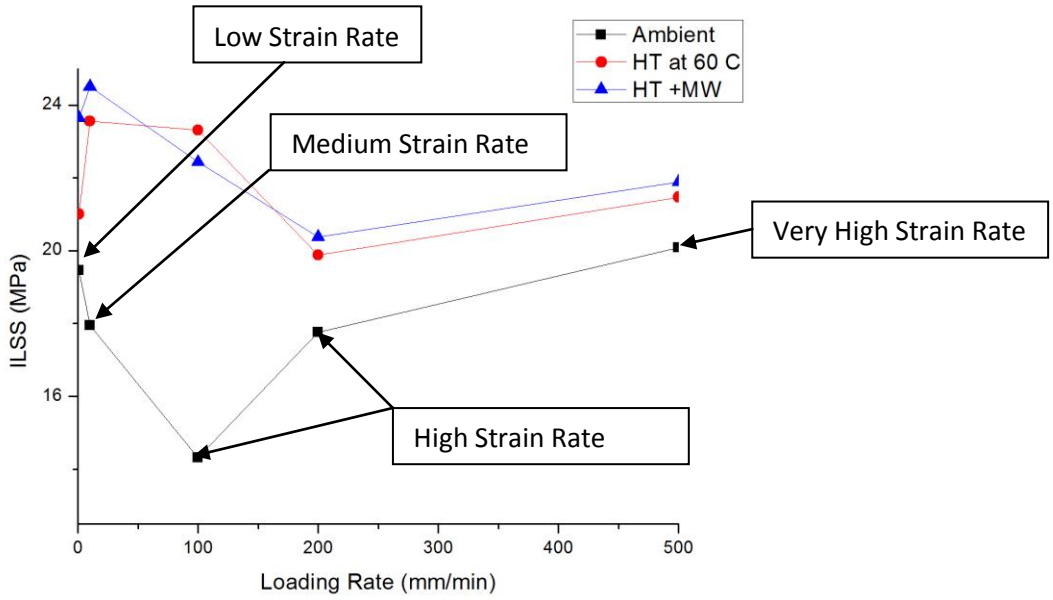


Fig 4.5: ILSS Vs Crosshead speed for Glass/epoxy composites at -80^{o}C

Alternatively,

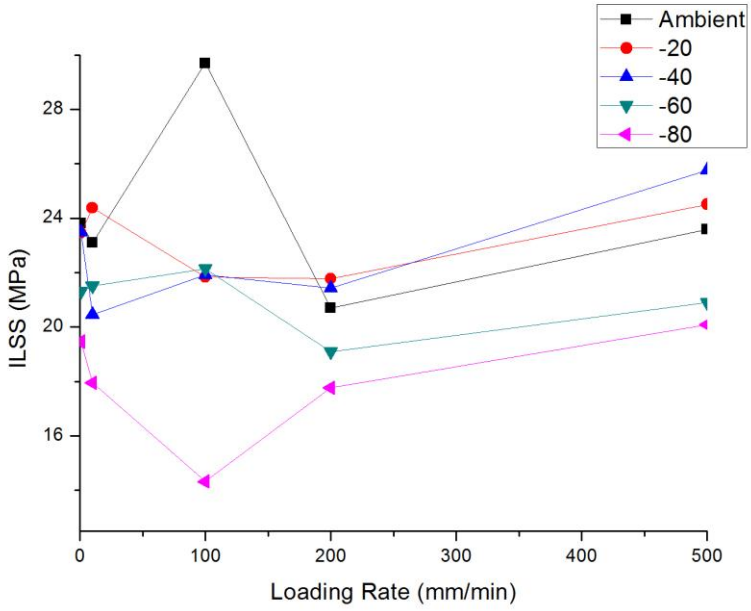


Fig 4.6: The ILSS values under Ambient Conditions at varying temperatures

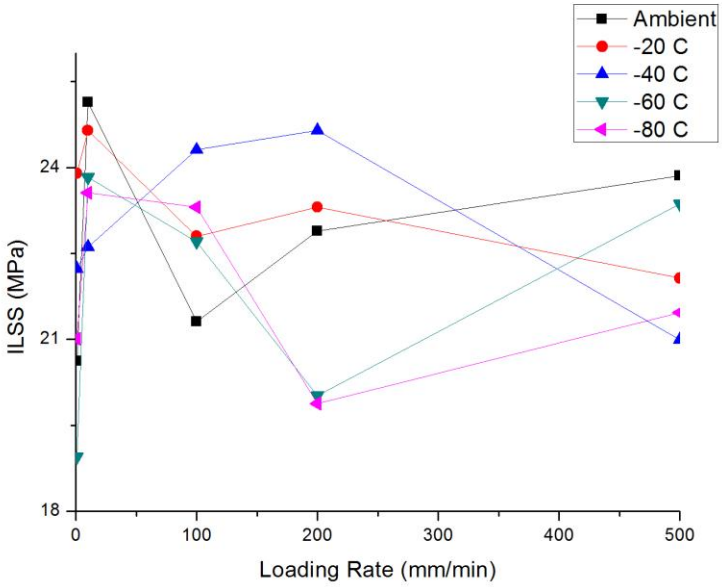


Fig 4.7: The ILSS values under Heat treated conditioned at 60^{o}C at varying temperatures

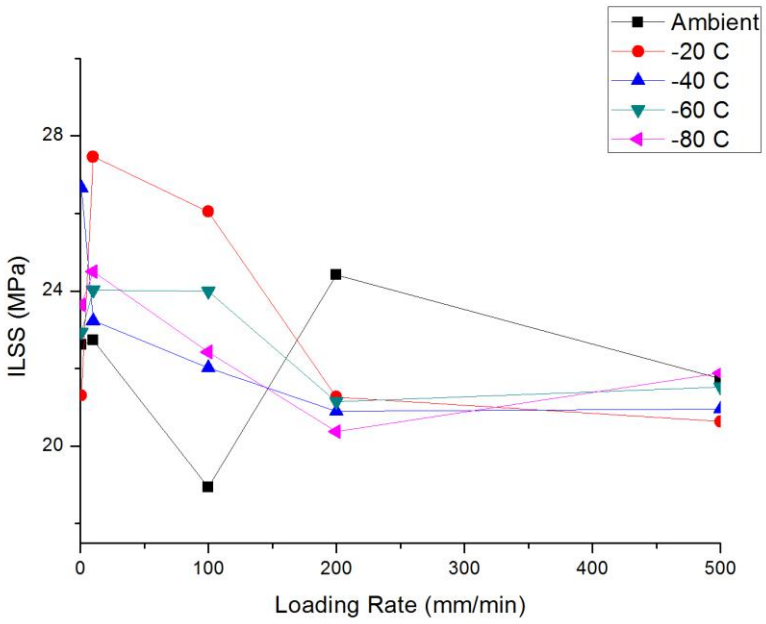


Fig. 4.8: The ILSS values under Microwave post Heat treated conditioned at 60°C at varying temperatures

4.1.2 Strain at Failure

Table 4.9 Strain at peak For Ambient Conditions:

Loading rates	Ambient	Heat treated at 60°C	Microwave post heat treatment
1	0.0293	0.0255	0.0282
10	0.0289	0.0302	0.0252
100	0.0079	0.0096	0.0063
200	0.0146	0.0162	0.0163
500	0.0343	0.0426	0.0365

Table 4.10 Strain at peak At-20^{0}C

Loading rates	Ambient	Heat treated at 60^{o}C	Microwave post heat treatment
1	0.0226	0.0293	0.0299
10	0.0289	0.0277	0.0306
100	0.0103	0.007	0.0142
200	0.0152	0.0135	0.0059
500	0.0692	0.0873	0.0388

Table 4.11 Strain at peak At-40 ^{0}C

Loading rates	Ambient	Heat treated at 60^{o}C	Microwave post heat treatment
1	0.0294	0.0305	0.0305
10	0.0279	0.0291	0.0279
100	0.0076	0.007	0.0066
200	0.0195	0.0168	0.0145
500	0.0452	0.0347	0.1134

Table 4.12 Strain at peak At-60 ^{0}C

Loading rates	Ambient	Heat treated at 60^{o}C	Microwave post heat treatment
1	0.0263	0.0294	0.027
10	0.0246	0.0314	0.0276
100	0.0067	0.0085	0.0099
200	0.0135	0.0147	0.0126
500	0.0422	0.0415	0.0356

Table 4.13 Strain at peak At -80 ^{0}C

Loading rates	Ambient	Heat treated at 60°C	Microwave post heat treatment
1	0.025	0.0252	0.0285
10	0.024	0.0231	0.0269
100	0.0066	0.0079	0.0099
200	0.0111	0.0173	0.0165
500	0.0512	0.0392	0.0962

Table 4.14 Strain at peak At varying Temperatures in ambient conditions:

Loading Rate	Ambient	-20°C	-40°C	-60°C	-80°C
1	0.0293	0.0226	0.0294	0.0263	0.025
10	0.0289	0.0289	0.0279	0.0246	0.024
100	0.0079	0.0103	0.0076	0.0067	0.0066
200	0.0146	0.0152	0.0195	0.0135	0.0111
500	0.0343	0.0692	0.0452	0.0422	0.0512

Table 4.15 Strain at peak At varying Temperatures Heat Treated condition at 60°C:

Loading Rate	Ambient	-20°C	-40°C	-60°C	-80°C
1	0.0255	0.0293	0.0305	0.0294	0.0252
10	0.0302	0.0277	0.0291	0.0314	0.0231
100	0.0096	0.007	0.007	0.0085	0.0079
200	0.0162	0.0135	0.0168	0.0147	0.0173
500	0.0426	0.0873	0.0347	0.0415	0.0392

Table 4.16 Strain at peak at varying Temperatures Microwave Heating post Heat Treated condition at 60 o C:

Loading Rate	Ambient	-20 oC	-40 oC	-60 oC	-80 oC
1	0.0282	0.0299	0.0305	0.027	0.0285
10	0.0252	0.0306	0.0279	0.0276	0.0269
100	0.0063	0.0142	0.0066	0.0099	0.0099
200	0.0163	0.0059	0.0145	0.0126	0.0165
500	0.0365	0.0388	0.1134	0.0356	0.0962

The corresponding graphs are as follows:

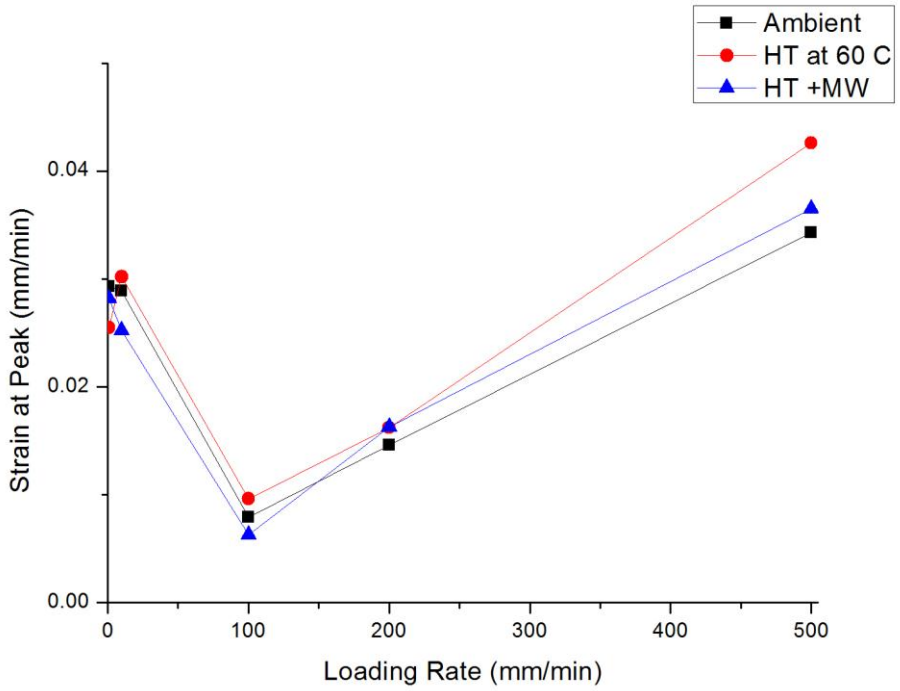


Fig 4.9: Strain Rate Vs Crosshead speed for Glass/epoxy composites at Ambient Conditions

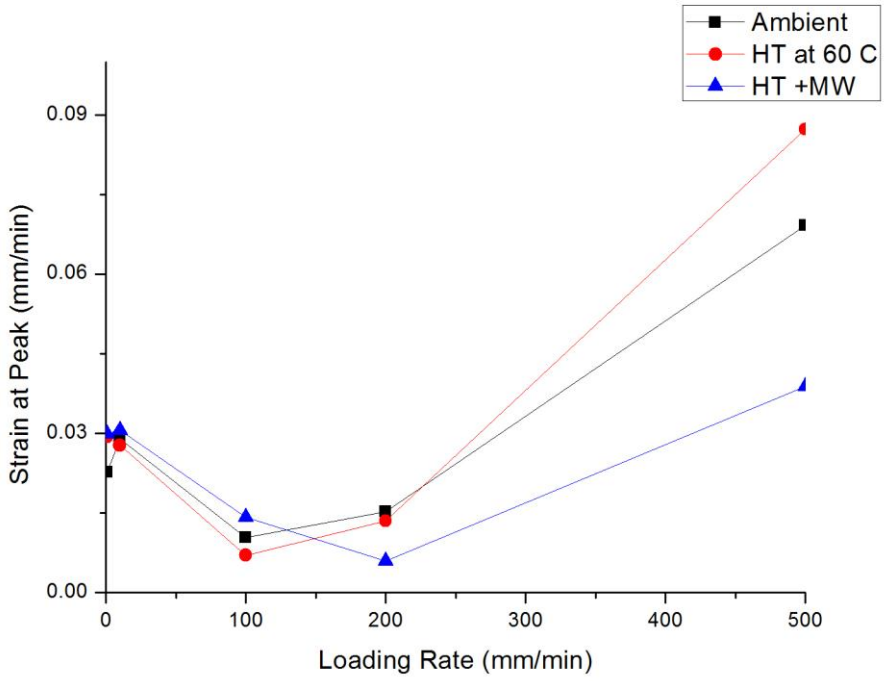


Fig 4.10: Strain Rate Vs Crosshead speed for Glass/epoxy composites at -20^{0}C

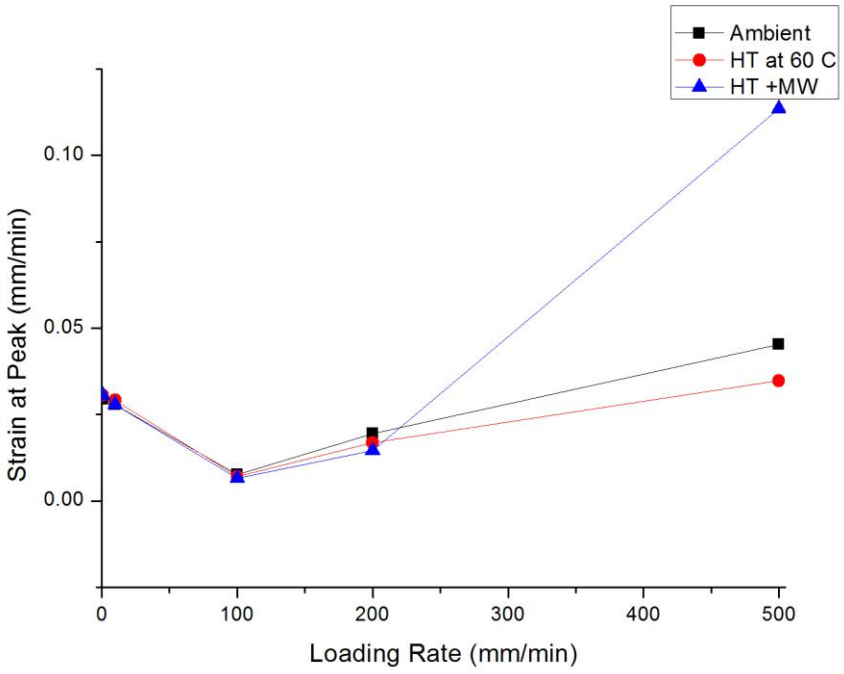


Fig 4.11: Strain Rate Vs Crosshead speed for Glass/epoxy composites at -40^{0}C

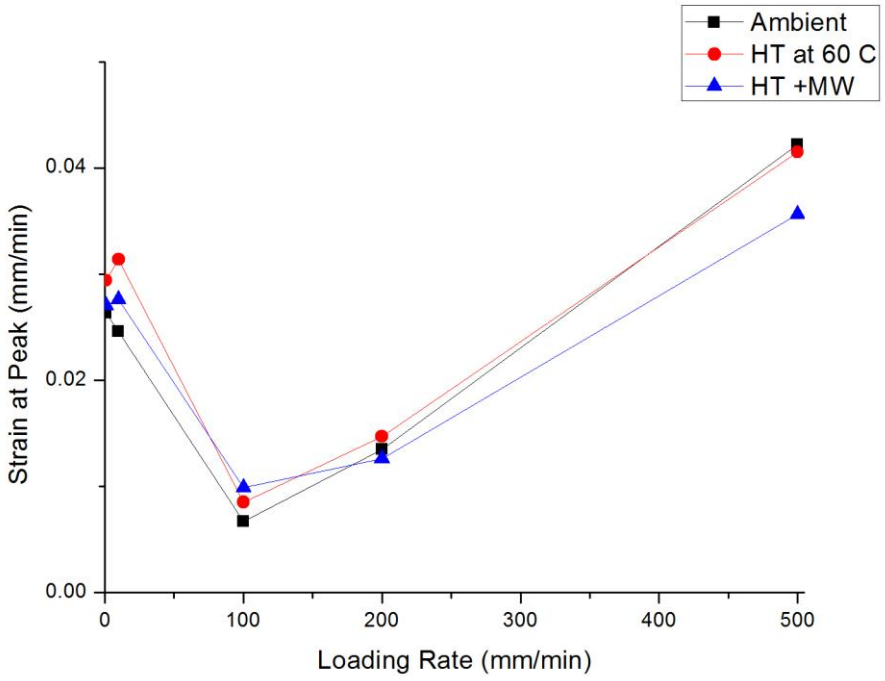


Fig 4.12: Strain Rate Vs Crosshead speed for Glass/epoxy composites at -60^{0}C

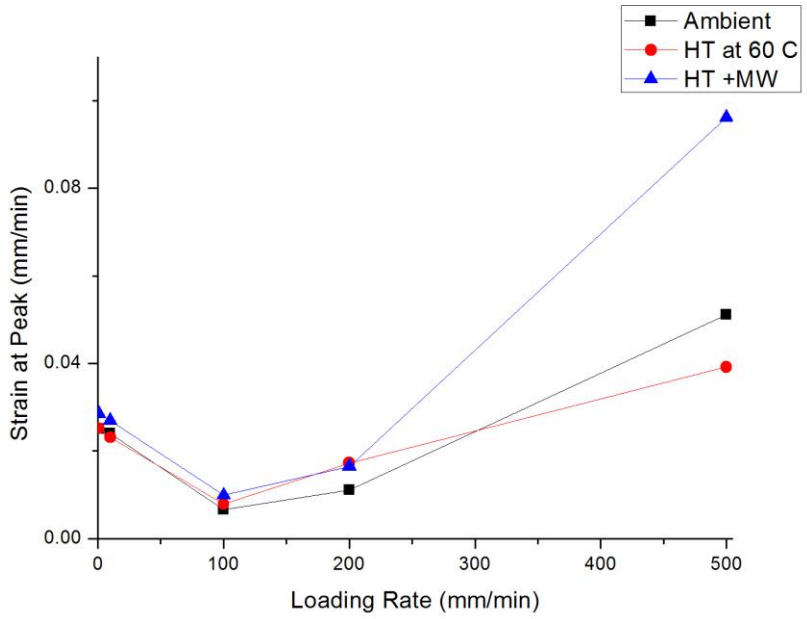


Fig 4.13: Strain Rate Vs Crosshead speed for Glass/epoxy composites at -80^{0}C

Alternatively,

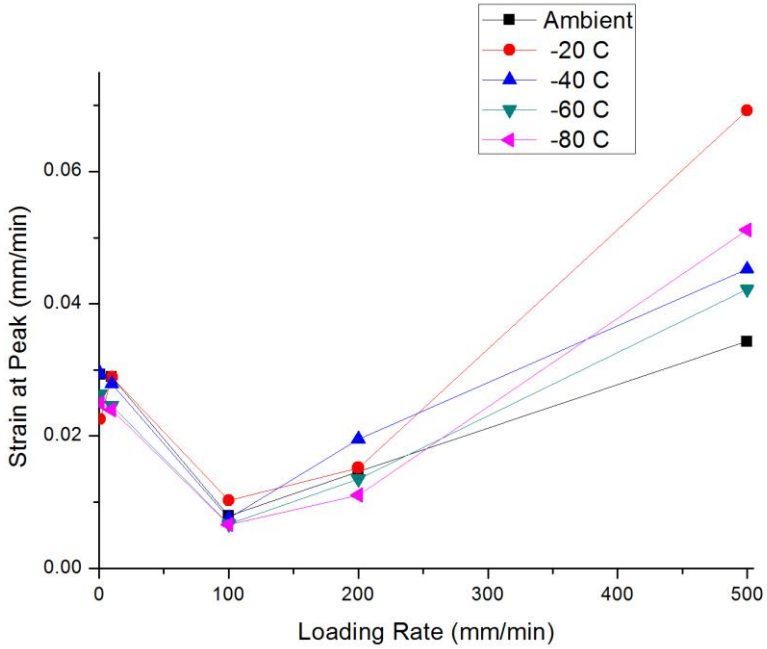


Fig 4.14: Strain Rate Vs Crosshead speed for Glass/epoxy composites in ambient preconditioning

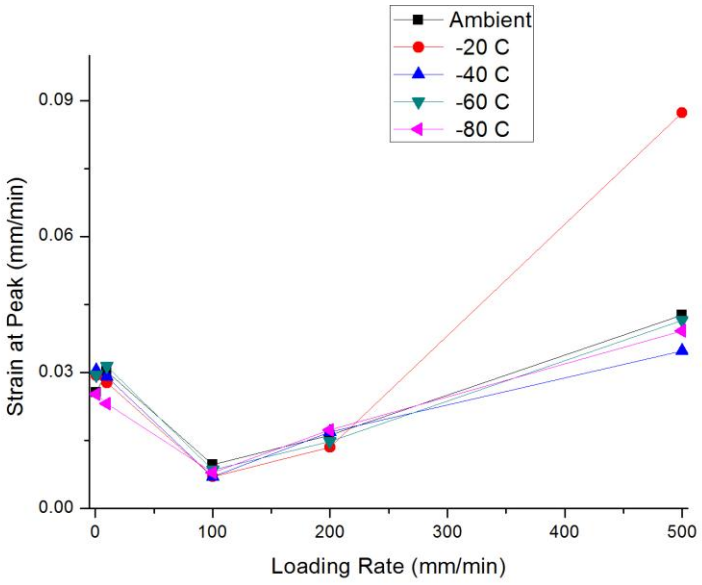


Fig 4.15: Strain Rate Vs Crosshead speed for Glass/epoxy composites in Heat Treated at 60^0C for 1 hour preconditioning

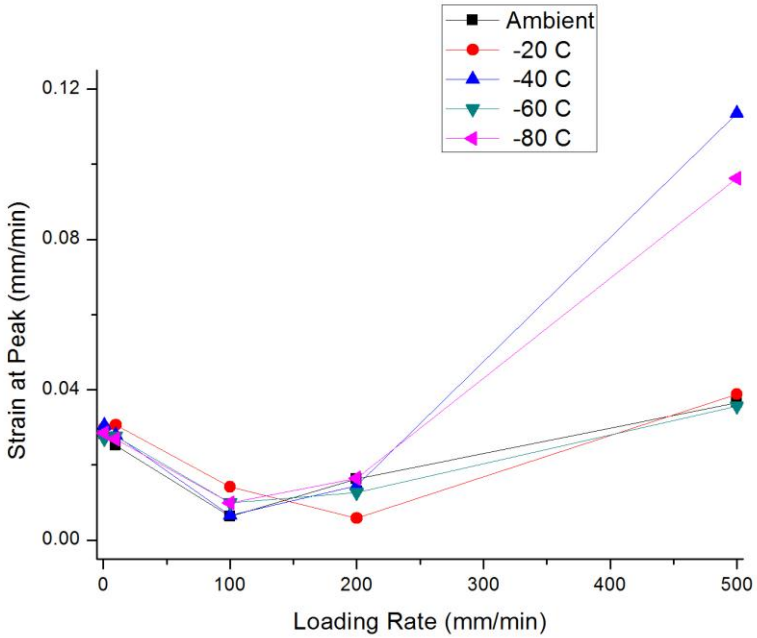


Fig 4.16: Strain Rate Vs Crosshead speed for Glass/epoxy composites in Microwave heating post Heat Treatment at 60^0C for 1 hour preconditioning

The graphs in fig.4.1-4.5 show the effect of pretreatment on the laminar composite. It is quite evident from the graphs that the composites with Microwave heating post Heat Treatment at 60^0C for 1 hour preconditioning possess the maximum ILSS values than that of thermal conditioning at 60^0C for 1 hour, followed by no pre-treatment (ambient).This phenomenon may be due to enhanced penetrating and semi penetrating network leads to further cross linking due to enhanced adhesion at the interface. Also, the microwave exposure may lead to penetration of two independent networks over each other without being covalently bonded. The mixture of this two independent network is called interpenetrating network (IPN). This delays the transition from glassy to rubbery state and thus makes the composite stronger. The graphs in fig.4.6-4.8 depict the effect of the different working temperatures at different loading rates under the same pre-treatment. Here, we can infer from these graphs that at low temperatures the composites exhibit lower values of Inter Laminar Shear Stress. This implies low strength possessed by the composites at lower temperatures. The possible reasons for this phenomenon may be hardening of matrix at low temperature. This may result to enhanced matrix cracking and debonding. The difference in thermal coefficients of the fiber and matrix may also be one of the reasons. The graphs in fig 4.9-4.13 show the variation of strain at failure. These curves give an overview on the impact of preconditioning, service temperatures, and loading speed on the glass fiber reinforced composites. By careful study of the curves, one can predict that the strength of the composite is increasing as we precondition it with thermal treatment, and further, microwave treatment enhances the strength of the composite to a higher degree. This is in agreement of

the ILSS values and graphs, and thus may be due to the aforesaid reasons. Moreover, the strain curve deciphers an increase in strain at failure, and supported by decrease in ILSS values at very high loading rate, when treated under microwave radiations post heat treatment. The possible reason for the phenomenon may be due to more compact structure of the polymer due to enhanced cross linking. Heat is generated as the composite is exposed to very high loading rate, but, there is very less scope for removal of heat. This slow rate of heat dissipation leads to higher amount of heat accumulated at the crack tip. Thus, the chances for breakage are most likely at the crack tip. This is in agreement with the corresponding ILSS values Also, the graphs in fig.4.14-4.16, we can predict about the behaviour of the composites at different temperatures. As explained above, the hardening of matrix can be the reason for the high strain rates and thus low strengths at lower temperatures.

4.2 Failure Analysis by SEM:

The Scanning Electron Micrographs revealed different modes of failure at low temperature in different conditions and at varying crosshead speed. The majority of them being as follows:

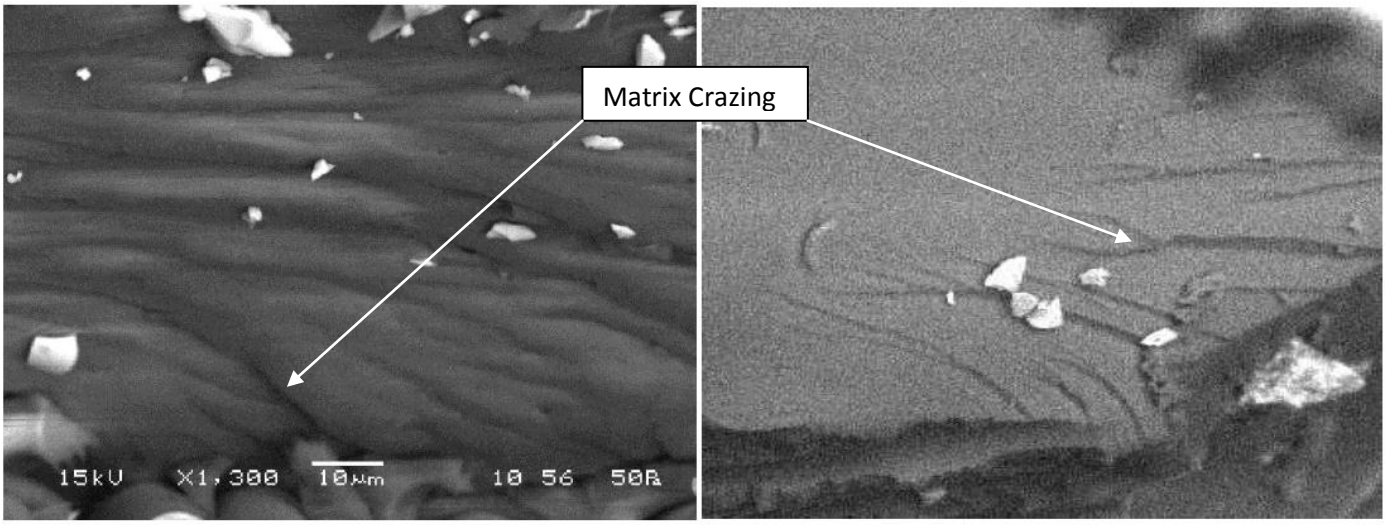
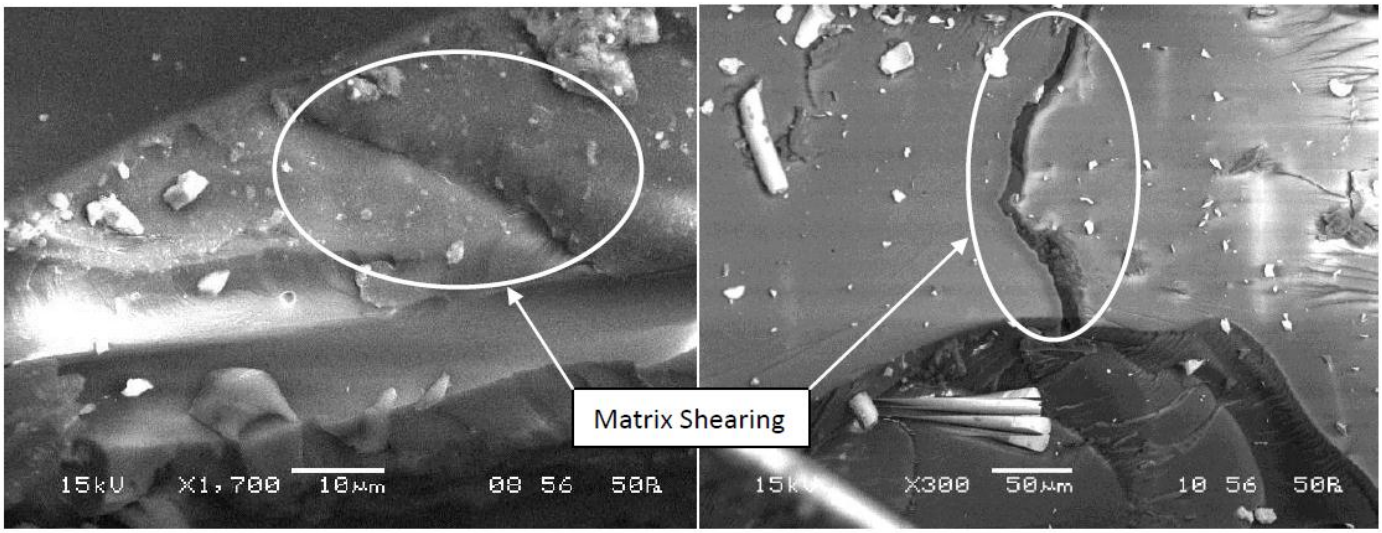
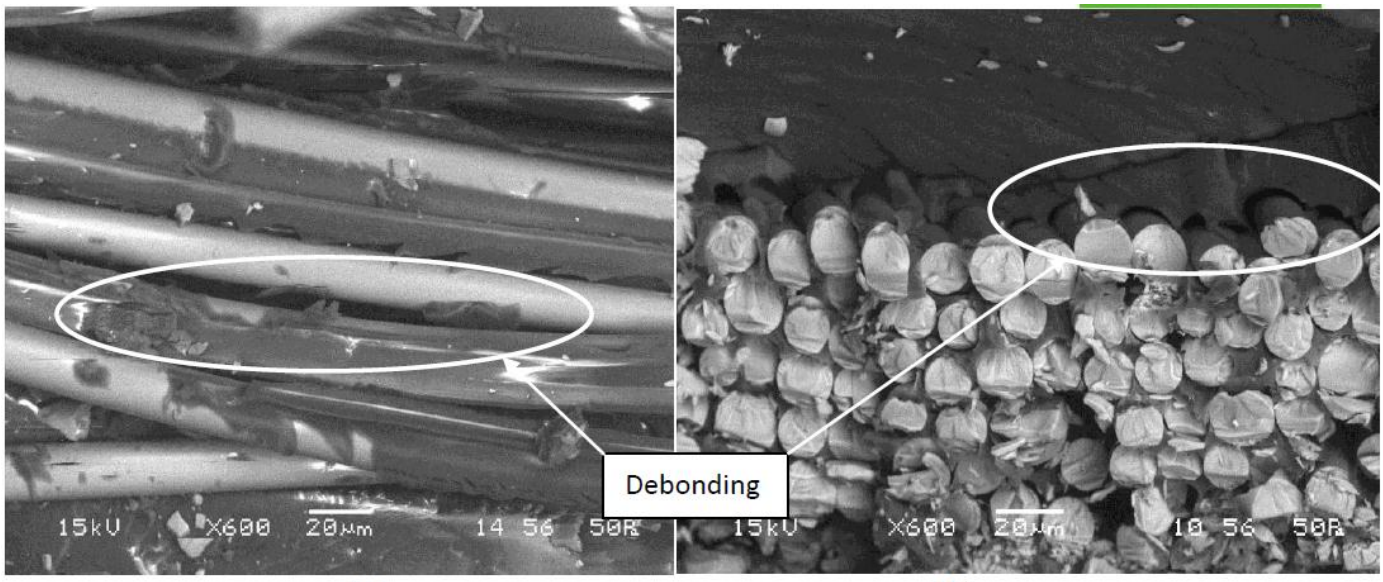


Fig 4.17 *Scanning Electron Micrograph shows matrix crazing having river like images*



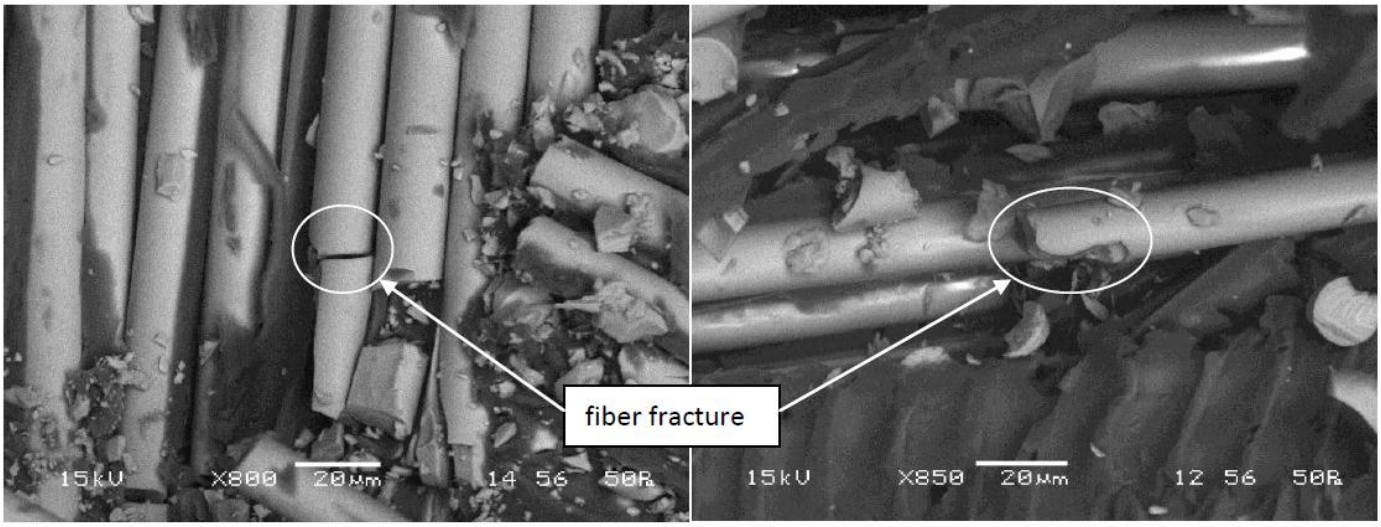
(a) (b)

Fig 4.18 *Scanning Electron Micrograph shows matrix shearing and matrix cracking*



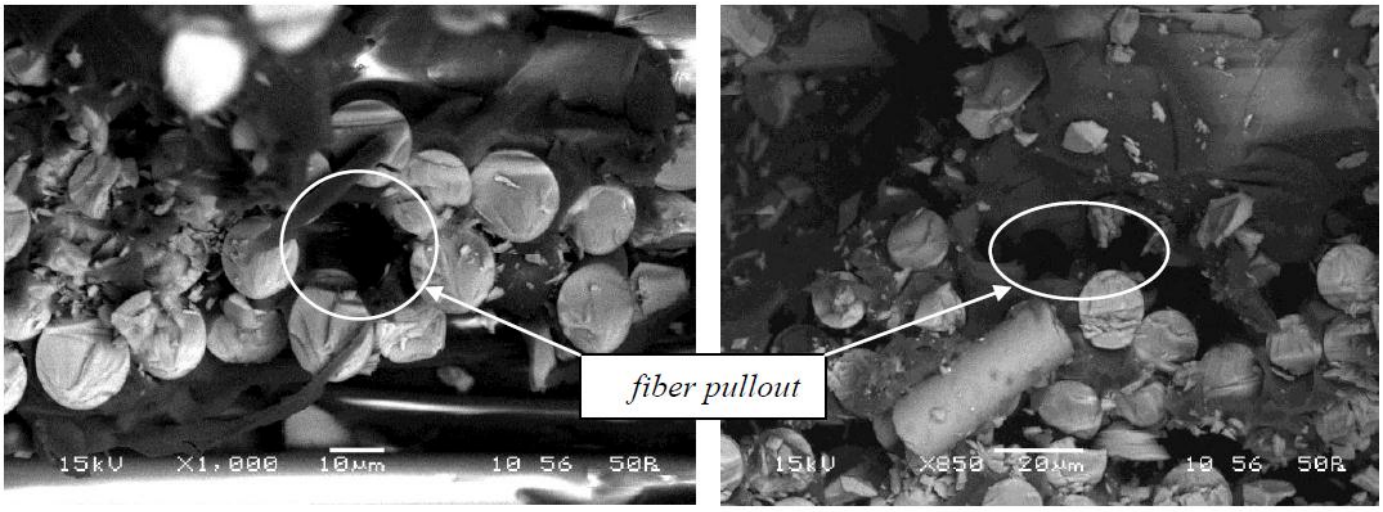
(a) (b)

Fig 4.19 *Scanning Electron Micrograph shows fiber-matrix debonding*



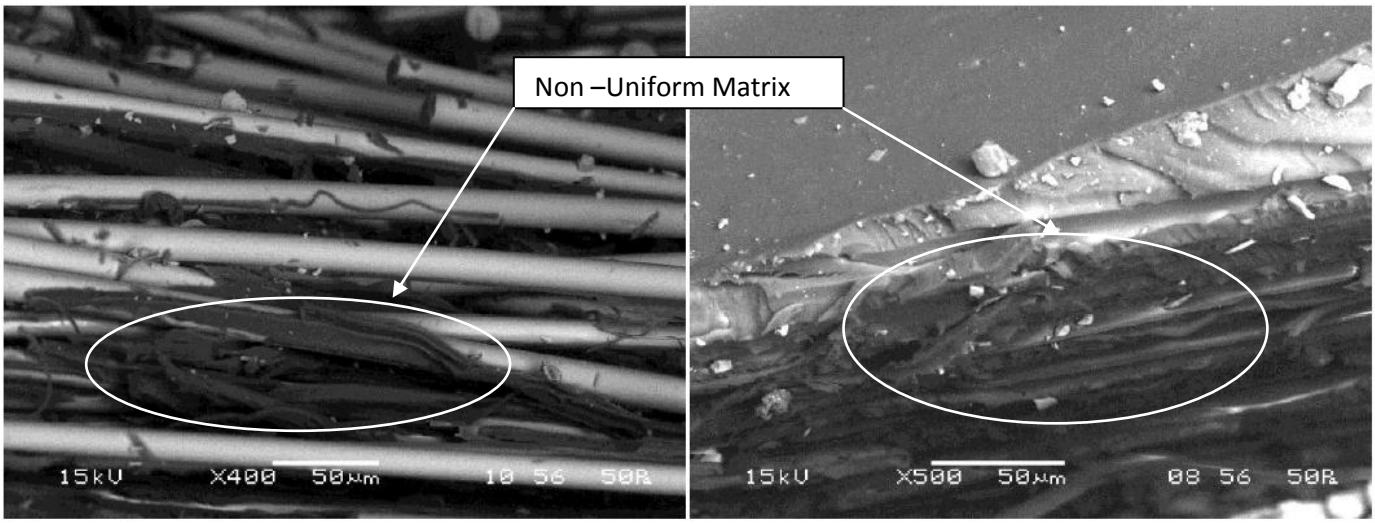
(a) *(b)*

Fig 4.20 *Scanning Electron Micrograph shows fiber fracture*



(a) *(b)*

Fig 4.21 *Scanning Electron Micrograph shows fiber pullout*



(a) *(b)*

Fig 4.22 *Scanning Electron Micrograph shows non-uniform matrix*

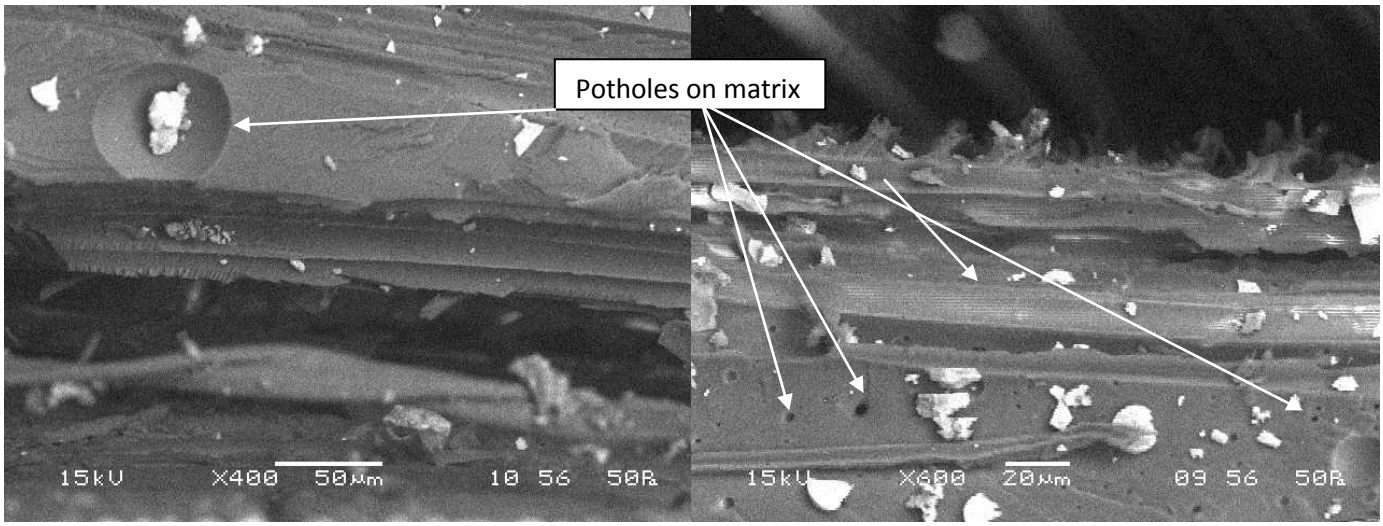


Fig 4.23 *Scanning Electron Micrograph shows Potholes created at low temperatures*

Large thermal alterations may lead to large amount of contraction or mismatch at the interface due to the inherent difference between thermal coefficient of fibres and polymer. This may lead nucleation of fiber matrix debonding at the fiber- matrix interface. At higher cross head speed, matrix crazing were visible that lead to the possible direction of propagation of the prominent mode of failure at that part of the composite. Fiber fracture, fiber pull out, matrix cracking and de-lamination serve to be the dominating modes of failure at higher rates of loading speed, which absorb energy and thereby increase the fracture toughness of the fibrous composite, and does not allow it to fail catastrophically. Due to the treatment at ultralow temperatures, potholes are present at the failed surfaces, which provide opportunity for the matrix crack to nucleate and thus act as nucleation site for the fracture. Non uniform matrix visible in the micrographs reveals the high rate of crack propagation during failure. The micro-voids visible may be formed during curing or stresses generated due to difference in thermal expansion coefficient between the glass fibre and the epoxy matrix. Shearing of matrix may lead to reduction of its ability to absorb energy, and thus it is more prone to crack propagation. As there is sufficient amount of energy present in the desired direction, fibers are pulled out from the matrix, which is an effective energy absorbing mechanism.

4.3 FTIR Graphs:

For Ambient atmosphere:

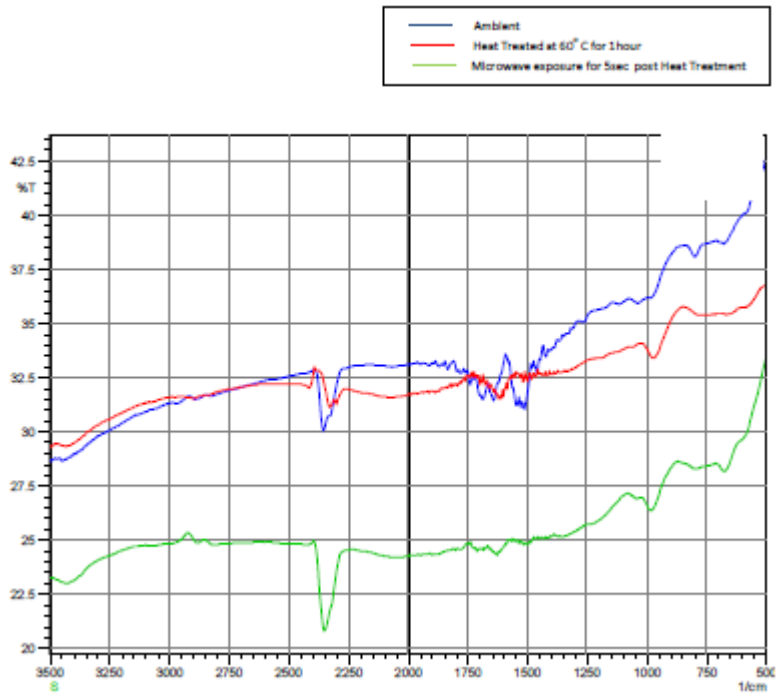


Fig 4.24: FTIR imaging for different pre-treatments at ambient Temperature

For -20^{0}C:

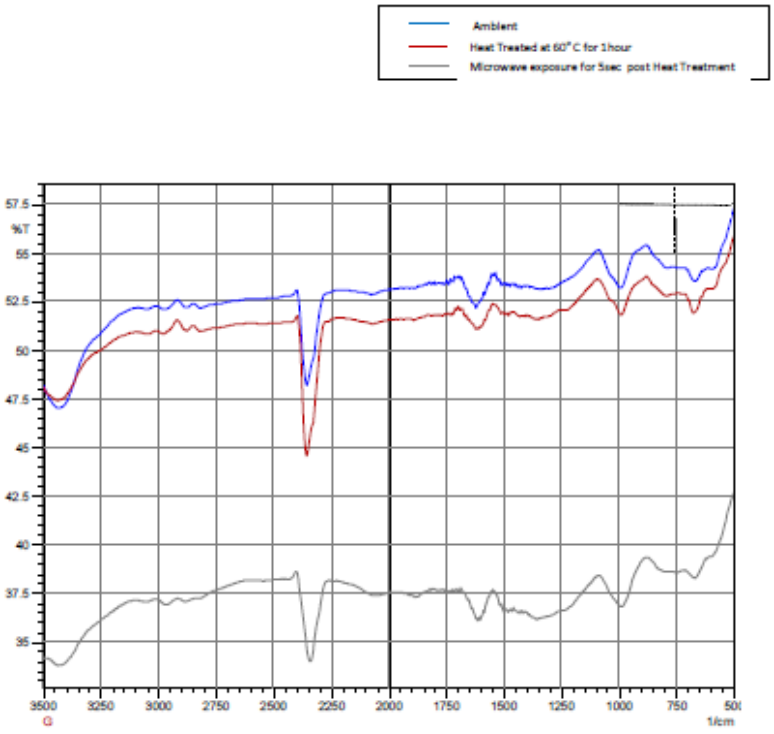


Fig 4.25: FTIR imaging for different pre-treatments at -20^{0}C

For -40^{0}C:

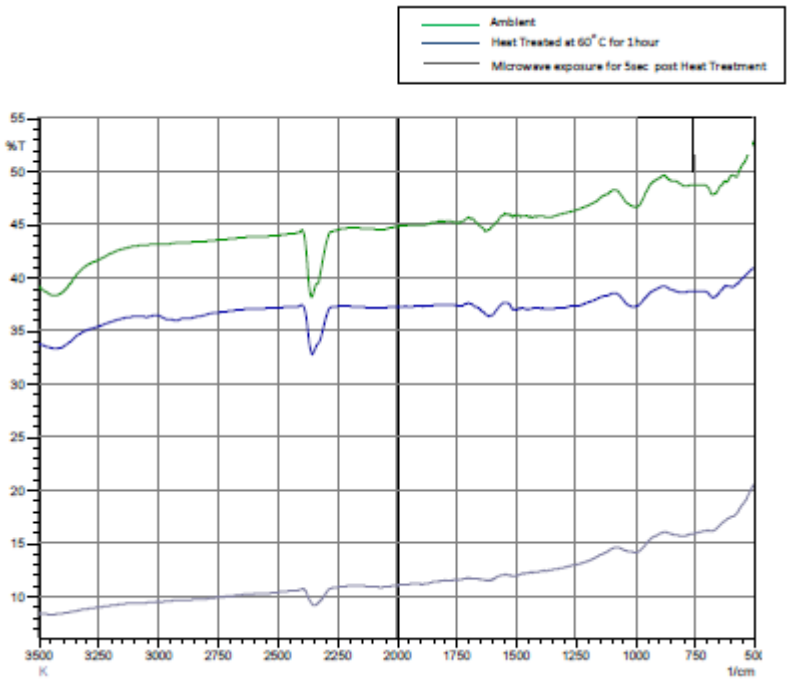


Fig 4.26: FTIR imaging for different pre-treatments at -20^{0}C

For -60^{0}C:

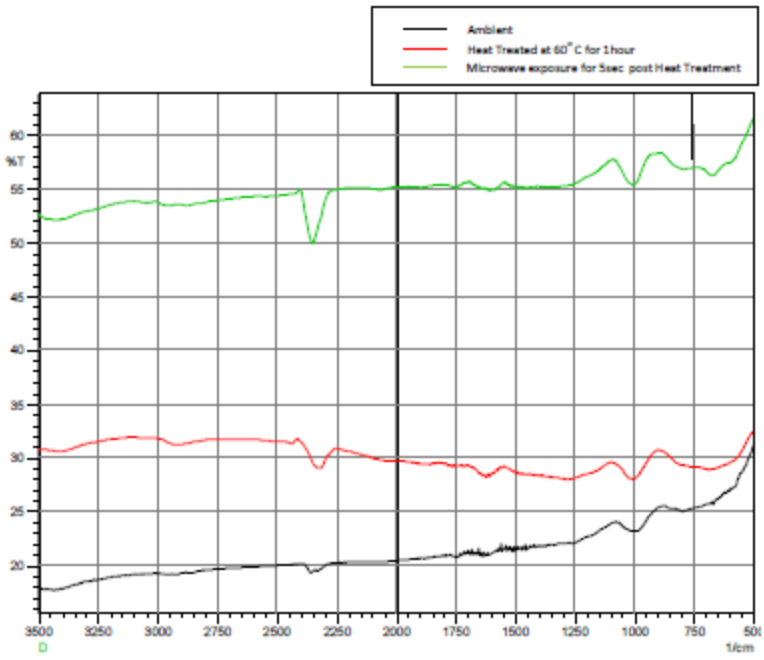


Fig 4.27: FTIR imaging for different pre-treatments at -60^{0}C

For -80^{0}C:

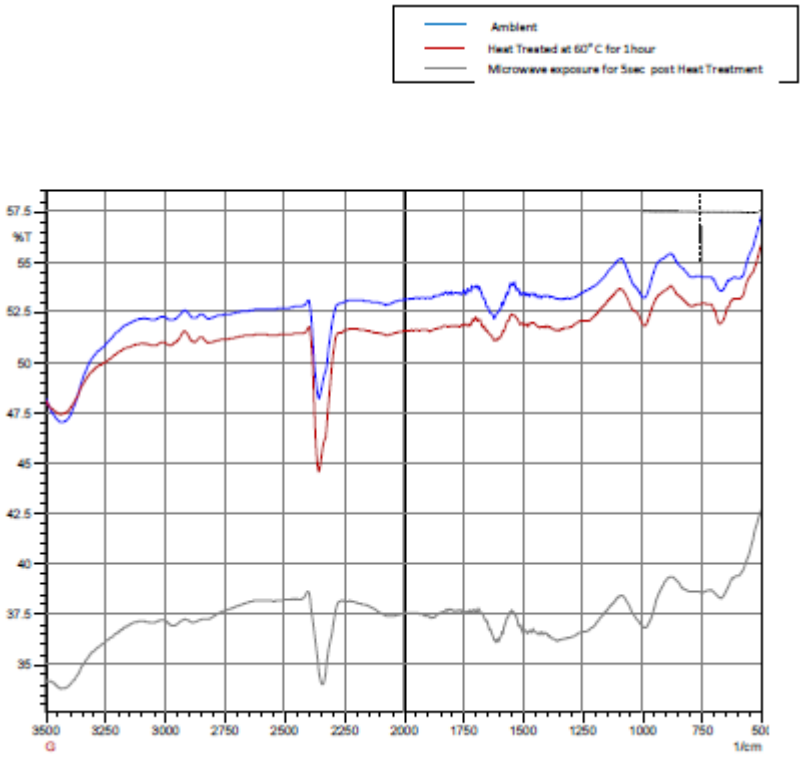


Fig 4.28: FTIR imaging for different pre-treatments at -80^{0}C

The FTIR Images show shift in spectra and thus give an idea about the extent of polymerization reaction that has occurred in the matrix of the composite due to different pretreatment and in different curves we can see the variations in different working temperatures. The curve representing the ambient conditioning acts as the reference for the rest of the two conditionings, viz., thermal aging at 60^0C for 1 hour, and exposure to microwave radiations for 5 seconds post heat treatment. Thermal treatment leads further polymerization due to higher degree of interfacial bonding. Thus the cross linking occurring in the polymer increases. When the composite is exposed to microwave radiations, homogenous heating results in drastic increase in polymerization. This is evident from the above graphs, as there is a huge shift in the curves representing microwave treated composites. The FTIR images depict the degree of polymerization as it shows the percentage of transmissivity with respect to wave number. Here it is observed, as it is thermally conditioned, the transmissivity decreases. This can occur possibly because of higher cross linking. Also, when it is followed by Microwave treatment, there is drastic decrease in the transmissivity values. This implies much high amount of polymerization due to pretreatment of microwave radiations.

4.4 Temperature Modulated Differential Scanning Calorimetry:

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For Ambient atmosphere:

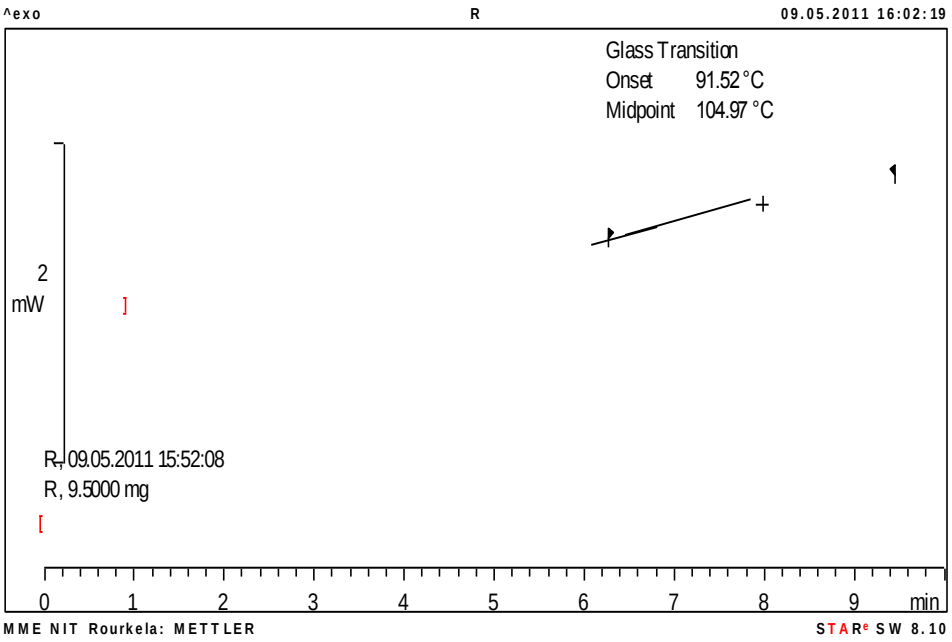

Fig 4.29: TMDSC Graph for ambient atmosphere

For -20^{0}C:

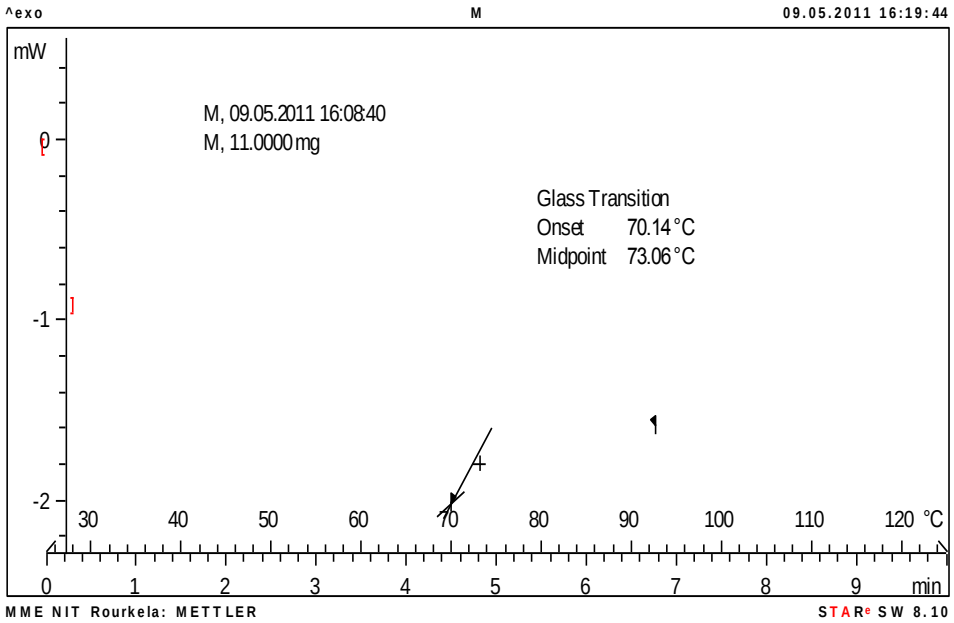

Fig 4.30: TMDSC Graph for -20^{0}C

For -40^{0}C:

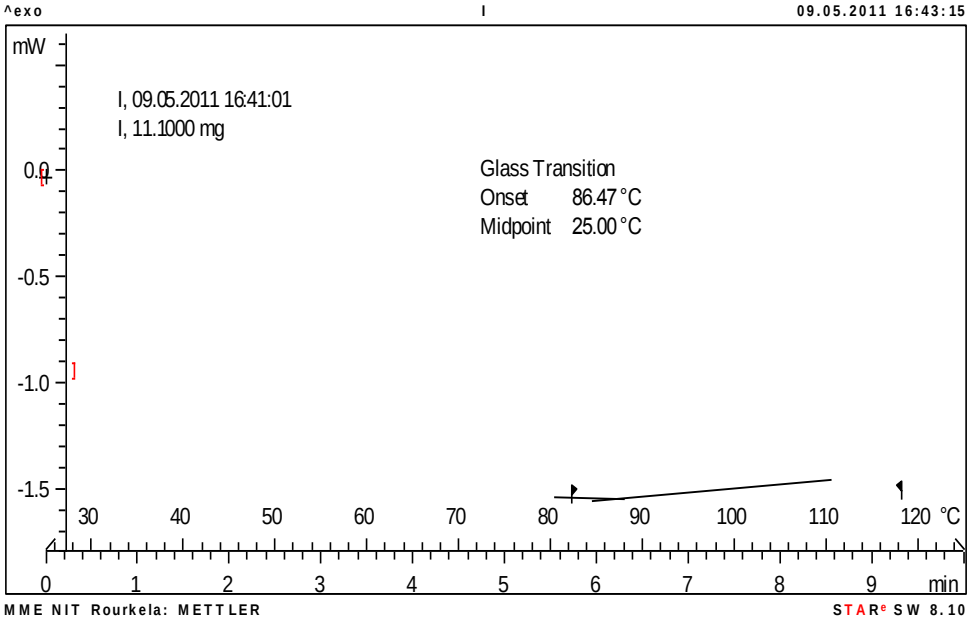

Fig 4.31: TMDSC Graph for -40^{0}C

For -60^{0}C:

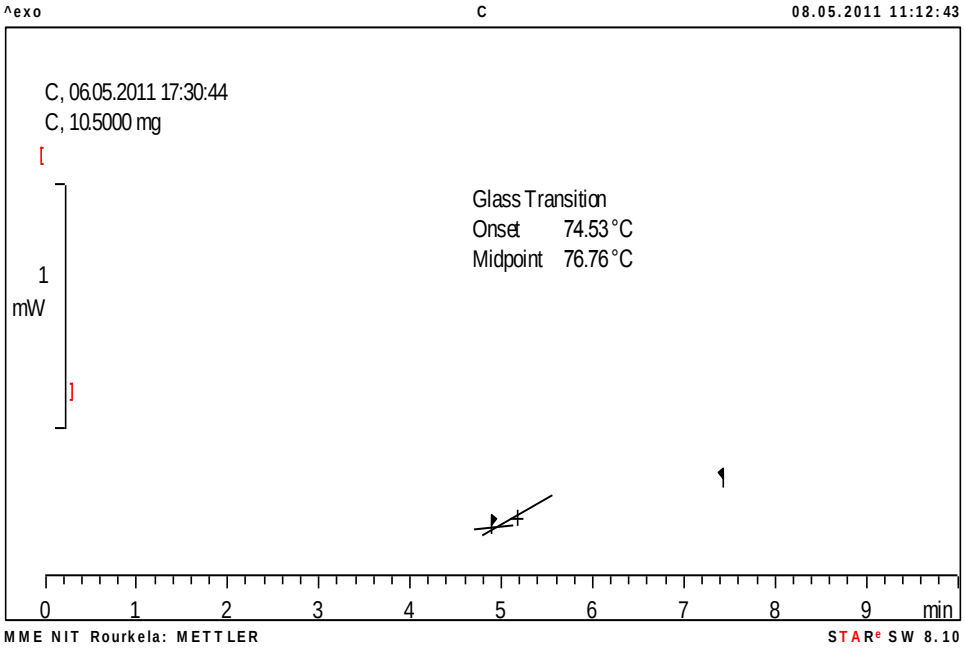

Fig 4.32: TMDSC Graph for -60^{0}C

For -80^{0}C:

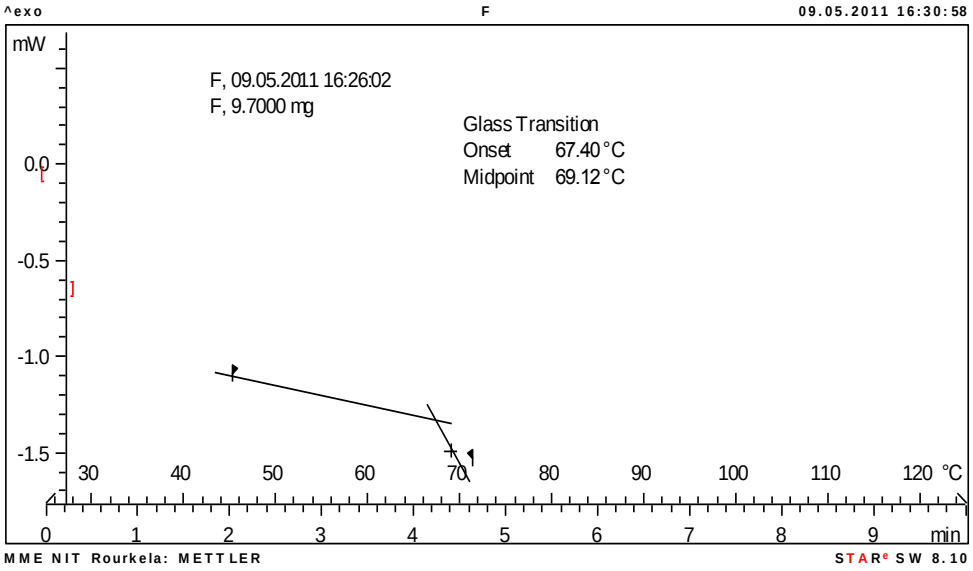

Fig 4.32: TMDSC Graph for -80^{0}C

A graph has been plotted to show the variation on Tg with respect to the conditioning temperature under ambient conditions.

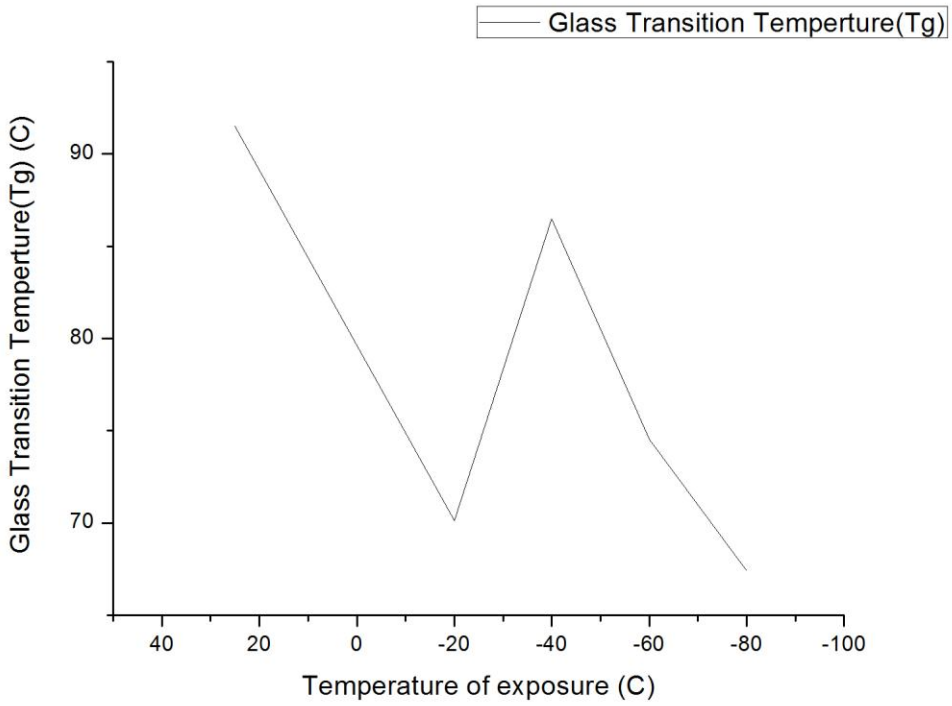

Fig 4.32: Variation of Tg with respect to the conditioning temperature under ambient conditions.

The Glass transition temperature,Tg, is a very important method as it gives an idea about the highest critical temperature that can be employed when the material is under engineering applications. Generally, a polymer is put into service much below its Tg. But when the composite is used at ultra low temperatures, the Tg decreases thereby resulting in very limited load bearing capacity. This phenomenon may be due to the embrittlement of the matrix and thus the interface is unable to transfer the load. This can limit the usage of Glass Fiber Reinforced Composite at ultra low temperatures. But in comparison to other materials , composites still are a challenging material for low temperature applications.

Chapter 5
Conclusion

Durability and long life of FRP composites under harsh and hostile environment have been major area of concern. The storage tanks, and bridges and walls used for railway lines in extremely cold countries like Russia suffer dual exposure of low temperature and load under varying rates. Composites can provide a solution for the material satisfying both these requirements. However, the heterogeneous nature of glass Fiber Reinforced Composite makes the process of its failure quite cumbersome. In the present research work, it is been tried to explain the failure mechanism possibly occurring in the tested samples on the basis of established theories through ILSS values, Strain at failure values, SEM fractographs, FTIR and TMDSC graphs. Endeavour has been made to estimate the micro and macro-mechanics of the possible modes of failures. The ILSS and strain at failure curves reveal the possible effect of different kinds of pretreatment By observing carefully the fracture surface of the composite as shown by the SEM micrographs, the factors affecting their respective failure could be determined.

FTIR images critically compare the effect of degree of polymerization under different working temperatures. The results of TMDSC suggest that there is a variation in the glass transition temperature and thus limits its usage under the given conditions. Summarizing all the above results, we may conclude that, thermal and microwave conditioning leads to enhanced polymerization and thus the matrix gets harder and also, there is enhanced interfacial property. From the results, it is deciphered that the usage of untreated composites is better than that of pretreated composites in low temperature conditions, as it can withstand more differential straining, and can thus absorb more energy, rendering enhanced service life. From the results, it is deciphered that the usage of untreated composites is better than that of pretreated composites in low temperature conditions, as it can withstand more differential straining, and can thus absorb more energy, rendering enhanced service life.

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